UNCLASSIFIED

AD NUMBER

ADA011845

NEW LIMITATION CHANGE

TO

Approved for public release, distribution unlimited

FROM

Distribution authorized to U.S. Gov't. agencies and their contractors; Administrative/Operational Use; 23 JAN 2009. Other requests shall be referred to CDR US Army RDECOM-ARDEC, Attn: AMSRD-AAR-MEE, Bldg 321, Picatinny Arsenal, NJ 07806-5000.

AUTHORITY

US Army RDECOM-ARDEC, Attn: AMSRD-AAR-MEE ltr dtd 16 apr 2009

THIS PAGE IS UNCLASSIFIED

UNCLASSIFIED

AD NUMBER

ADA011845

NEW LIMITATION CHANGE

TO

Distribution authorized to U.S. Gov't. agencies and their contractors; Administrative/Operational Use; 23 JAN 2009. Other requests shall be referred to CDR US Army RDECOM-ARDEC, Attn: AMSRD-AAR-MEE, Bldg 321, Picatinny Arsenal, NJ 07806-5000.

FROM

Approved for public release, distribution unlimited

AUTHORITY

US Army RDECOM-ARDEC, Attn: AMSRD-AAR-MEE ltr dtd 23 Jan 2009

THIS PAGE IS UNCLASSIFIED

ADA 011 845

ENCYCLOPEDIA OF EXPLOSIVES AND RELATED ITEMS

PATR 2700

VOLUME 6

BY

BASIL T. FEDOROFF & OLIVER E. SHEFFIELD

ASSISTED BY

THE FRANKLIN INSTITUTE RESEARCH LABORATORIES

U.S. ARMY RESEARCH AND DEVELOPMENT COMMAND
TACOM, ARDEC
WARHEADS, ENERGETICS AND COMBAT SUPPORT CENTER
PICATINNY ARSENAL
NEW JERSEY, USA
1974

The Franklin Institute Research Laboratories
Philadelphia, Pennsylvania 19103

Technical Contributions Provided by:

Mr Gunther Cohn, Senior Staff Engineer,
Applied Physics Laboratory

Dr Richard Mason, Senior Information Analyst
Science Information Services

Mr Daniel J. Mullen, Jr., Senior Research Engineer,
Applied Physics Laboratory

Dr J. Robert Thornton, Senior Information Analyst,
Science Information Services

Copies of the "Encyclopedia of Explosives
And Related Items" can be obtained by requesting CD ROM
from the:

National Technical Information Service
(formerly Clearinghouse)
US Department of Commerce
Springfield, Virginia 22151

1-800-553-6847 USA only
703-605-6000
www.ntis.gov/FCPC

The contents of these volumes are UNCLASSIFIED

The distribution of these volumes is UNLIMITED

Neither the US Government nor any person acting on behalf of
the US Government assumes any liability resulting from the use or
publication of the information contained in this document or warrants
that such use or publication will be free from privately owned rights.

All rights reserved. This document, or parts thereof, may not be
reproduced in any form without written permission of the
Energetics and Warhead Division, WECAC, TACOM, ARDEC, Picatinny Arsenal

Library of Congress Catalogue Card Number: 61-61759

PREFACE

This volume represents a continuing effort to cover comprehensively the unclassified information on explosives and related subjects in the same manner and format as in previous volumes. The reader is urged to obtain the previous volumes and to read both the PREFACE & INTRODUCTION in Volume 1 in order to understand the authors' way of presenting the subject matter

In preparation for and during the writing of this Encyclopedia, the authors have consulted freely with and have had the cooperation of many individuals who contributed their expert knowledge and advice. This fact is acknowledged throughout the text at the end of the subject item. A listing of many others who have helped in various ways would be impractical

Scientists at the Franklin Institute Research Laboratories, Philadelphia, Pennsylvania (See overleaf) have contributed significantly in the organizing, literature searching and writing of much of the manuscript for this volume

Dr R.F. Walker, Explosives Division Chief, has provided financial support and encouragement to continue this work to its conclusion. Further financial support is expected from the sale of copies to non-governmental agencies and individuals by the National Technical Information Service, US Department of Commerce, Springfield, Virginia 22151. This additional source of funds will permit future volumes to be published much sooner

Although considerable effort has been made to present this information as accurately as possible, mistakes and errors in transcription and translation do occur. Therefore, the authors encourage the readers to consult original sources, when possible, and to feel free to point out errors and omissions of important works so that corrections and additions can be listed in the next volume. The interpretations of data and opinions expressed are often those of the authors and are not necessarily those nor the responsibility of officials of Picatinny Arsenal or the Department of the Army

This report has been prepared for information purposes only and neither Picatinny Arsenal nor the Department of the Army shall be responsible for any events or decisions arising from the use of any information contained herein

TABLE OF CONTENTS

	Page
Preface	I
List of Figures and Illustrations	V
Supplement to the List of Books on Explosives and Propellants	X
Descriptive Text of Encyclopedia Items:	
Etagenguss to Eyring Detonation Theory	E142 to E508
"f" to FZG	F1 to F259
"G" to Gyroscopic Movement of Projectiles	G1 to G197
TABLES	
Composition of Alcohols Intended for Ordnance Use	E156
Chemical and Physical Requirements for Diethyl Ether	E186
Absorbance Limits for Platinum-Cobalt Stock Solution	E187
Refractive Indices of Pure Compounds	E268
Refractive Indices of Mixture of Ethylene Glycol Dinitrate and Nitroglycerin	E269
Effect of Varying Proportions on Refractive Indices of Mixtures of Ethyleneglycol Dinitrate and Nitroglycerin	E269
Refractive Indices of Pure and Dynamite Nitroglycerin	E269
Infrared Method of Determining EGDN in Mixtures with NG	E270
Absorptivity of Benzene and 2-Nitropropane	E271
Absorptivities of NG and EGDN	E271
Binary Eutectic Mixtures of Explosives	E344
Ternary Eutectic Mixtures of Explosives	E346
Pyrodialytes	E366
Perchlorate	E367
Explosion Temperatures and Times for Various Explosives	E405
Activation Energies of Various Explosives	E406
Relative Performance Parameters of Underwater Explosives	E429
Explosive Switches of XM 60 Series	E449
Measured Properties and Calculated Parameters of Representative Cratering Explosives	E470
Table of US Specifications	E483
Explosives Consumption	E489
French Poudres Favier	F10
French Explosifs Favier	F11
Tests of Noninitiating Explosives	F23
Bichel's Flame Measurements Using 100g Samples of Explosives	F46
Portable Type Flame Projectors	F55
Composition of Flamethrower Mixtures – Gel Type	F59
Flammivores (Belgian Permissible Explosives)	F62

TABLES (Contd) **Page**

Characteristics of Various Illuminating Flares	F65
Hand-Manipulated Signal Devices	F66
Energy Release Tests	F101
Cylinder Test Results	F101
FNH Single-Base Propellants	F152
Calculated Thermochemical Values for US Propellants	F157
Percentage Products of Explosion for Explosives	F158
Belgian Forcites	F161
Forcites Antigrisouteuses	F162
Fördits	F162
Fractorites	F179
French HE Service Bombs of WWII	F189
French Dynamites-gommes	F192
French Dynamites-gélatinées	F192
French Dynamites grisouteuses	F193
French Weapons of WWII	F200
French WWII Mines	F201
Friction Compositions for Friction Detonators	F202
Fuel Gas Sources	F210
Priming Compositions Containing Mercury Fulminate	F230
Fumigène Compositions	F239
Properties of Spencer Chemical Company AN Prill Explosives	G14
Composition of Gases Produced on Detonation of Explosives	G22
Composition of Gases Produced on Detonation of Tetranitromethane	G22
Compression Values of Gasless Explosives	G26
Calculated Total Detonation Pressures of Gases Produced by Explosives	G31
Calculated Total Detonation Pressures Reported by Gorst	G32
Experimental Detonation Pressures Reported by Davis	G32
Experimental Gas Volumes Produced on Detonation of Explosives	G37
Sample Calculation of Gas Pressure Test Data	G41
Composition of Gases in Apparatus after Test	G41
Composition of Water Gas Reported by Riegel	G41
Gelatine-Carbonit Dynamites	G46
Gelatine-Dahmenit Dynamites	G47
Gelatine Dynamites	G48
Gelatine-Penthrinits and Penthrinits	G52
Gelatine Prosperits	G52
Gelatine Tremonits	G53
Gélatines, Vergé	G53
Single-Dose Oral Toxicities of Various Glycols and Related Compounds	G113
Properties of Graphite Used in Ammunition	G128
Solubility of Nitroguanidine in Aqueous Sulfuric Acid	G155
Specification Requirements for Nitroguanidine	G162
Characteristics of V2 Rocket	G184
List of US Artillery Weapons	G188
Gun Propellants, Advanced	G194

LIST OF FIGURES AND ILLUSTRATIONS

	Page
Titration Flask Assembly	E159
Typical Chromatograph of US Industrial Chem Co, NY, Filmex C	E161
Gas Chromatographic Scan of a Final Dried Single-Base Powder Sample	E165
Solution Tube	E166
Vacuum Line Assembly	E166
Wire Screen Tray	E166
Protective Drying Tube	E167
Solution Tube Holder	E167
Counterpoise	E167
Block Diagram of Electrolytic Hygrometer	E170
Distillation Times for Moisture Determination in Propellants and Explosives	E172
Apparatus for Estimation of Ethanol and Ether in Propellants	E175
Berl Pipette	E197
Kipp Generator and Storage System	E212
Titration Flask	E212
Ethoxy Apparatus	E221
Infrared Curve for 1-Nitro-2-propanol	E274
Infrared Curve for Ethyleneglycol Dinitrate with 0.25% 2-NPA	E275
Infrared Curve for Propellant	E276
Initiation or Explosion Temperature Determination	E277
Bottle and Metal Carrier for Ethylene Oxide Determination	E286
Ethylene Oxide Propellant Sampler	E289
Explosion Suppression System	E387
Power Producing Explosive Assembly	E394
Diagram of Modified Explosion Temperature Apparatus	E404
Explosive Diode	E406
Instant Explosive Excavation Pattern	E409
Explosive Bullet Caliber .50, T1	E411
Explosive Bullet Caliber .30, T1	E412
Explosive Incendiary Bullet Caliber .50, FA T1	E413
Explosive Incendiary Bullet Caliber .50, FA T2	E414
Conventional vs Explosive Stress Rates	E416

LIST OF FIGURES AND ILLUSTRATIONS (Contd) **Page**

Explosively Formed Jet-Motor Housing E416

Basic Explosive Forming Method E417

A Simple Arrangement for Explosive Forming E417

Explosive Sizing of Cones or Cylinders E421

Linear Array of Shot and Gage Holes for Strain Measurements E426

Detail of Linear Array Test E427

Underwater Test Setup E428

Explosive Pipe Closure System E430

Cross-Sectional View of a Single-Piston Press E431

Cross-Sectional View of a Double-Piston Press E431

Explosive Rivet E443

Explosive Switch Mk 66 Mod 0 E449

Contact Arrangement of XM 61 Switch E449

Explosive Switch Mk 46 Mod 0 E450

Explosive Actuator Mk 3 Mod 0 E450

Top Initiation for Explosive Welding E452

Furcated or "T" Configuration Explotron E492

"Y" Configuration Explotron E492

Tellurian (Underground) Explotron E494

Improved Sausage Machine E501

Macaroni and Blocking Presses E502

Extrusion Press E503

Faustpatrone F9

Field Proof Gun F22

US Standard Firing Pin for Stab Initiators F36

Stab Detonator, M55 F36

Percussion Primer, M29A1 F37

Percussion Pin for Bomb Fuze, M904, to Initiate M9 Delay Element F37

Rotating Drum Apparatus for Measuring Flame Characteristics Produced on Explosion F48

Rotating Drum Apparatus and Motion Picture Method F49

German Flange Projectile G63

Aircraft Parachute Flare, Mk5 F68

Signal Flare, M11 F69

LIST OF FIGURES AND ILLUSTRATIONS (Contd)

	Page
Signal Flare, M53A2–M58A2	F69
German Flares	F71
Magicube (Flash Photography Without Battery)	F77
Cleveland Open Cup for Flash and Fire Points	F81
Tag Open Cup for Flash Point of Liquids	F82
Tag Closed Tester	F89
Pensky-Martens Closed Tester	F91
Test Cup	F92
Cover Proper	F92
Test Cup and Lid Assembly	F93
Thermometer, Adapter, Ferrule and Packing Plug	F95
Flechette Projectile	F102
Diagram of Bullet Rigidity and its Operation During Flight	F104
How the "Weathercocking Effect" Influences the Flight of a Flechette	F105
Comparison between a 10-grain Flechette and a 55-grain Remington Caliber .223 Bullet	F105
Fluidic Explosive Initiator	F112
Digital Interval Timer Block Diagram	F113
Schematic of Flueric Amplifiers	F114
Schematic of Flueric Pressure-Compensated Oscillator	F114
Schematic of Flueric Counter Stage	F115
Flueric Timer Packaged for Artillery Fuze	F116
Fuze Armed by Ram Air Pressure	F117
Projectile Inside Shaped Charge	F154
Benjamin Franklin's Method of Electrical Initiation	F185
Friction Pendulum Test Apparatus	F204
Friction Test Apparatus of Bowden	F205
Friction Tests for Liquid & Solid Explosives	F205
Torpedo Friction Test Apparatus	F205
Friction Apparatus of Bowden-Kozlov	F206
Fuel Gas Cell Operated on Hydrogen	F209
Mercury Fulminate Crystals	F220
Navy Light Mk 1 Mod 1 Fusee	F248
Safety Fuses	F253

LIST OF FIGURES AND ILLUSTRATIONS (Contd) **Page**

Construction of UERL Diaphragm Gauge G3

Construction of a Typical Size of SR-4 Bonded Strain Gage G4

Three Designs of Bonded Rosettes Used Where Stresses are Multi-Directional G4

Simple Form of Unbonded Strain Gage Developed by Statham Laboratories G4

Simplified Strain Gage Circuits G5

Strain Gages on Loading Screws Show Pressure between Calendar Rolls G5

Gaine No 13 G6

Gas Analysis Apparatus G16

Gas Cannon Device G17

Gaseous Discharge Tube G20

Details of Construction of Crawshaw-Jones Apparatus G39

Smoke Producing Generator G61

German Military Explosives Used as Fillers in Anticoncrete and Armor-Piercing Shells G66

Giles Flask and its Applications G74

Glass Bulbs for Weighing Acids G78

Glass Bulb for Sampling Acids G79

Apparatus for Determination of Nitrogen in Nitroglycerin G91

Flow Chart of Typical Acid Preparation Plant G101

Flow Diagram of Batch Nitroglycerin Process G102

Nitrometer Apparatus for Determination of Nitrogen in Nitroglycerin G105

Typical Shapes of Powder Grains G123

Lischin's Hand Grenade, Truncheon Type G135

Mill's Hand Grenade, Egg Type G135

Fragmentation Hand Grenade, M57 G136

HEAT Rifle Grenade, M31 G137

Grenade Launcher, M7A3 G140

Guidance Systems G175

Guided Missiles G182

FZG 76 "V-1" Flying Bomb G183

V-2 (A-4) Rocket G184

German Guided Missile Launchers G185

LIST OF FIGURES AND ILLUSTRATIONS (Contd) **Page**

German High Pressure Pump or Revenge Weapon-3 G189

German Wind Gun G190

SUPPLEMENT TO THE LIST OF BOOKS ON EXPLOSIVES AND PROPELLANTS GIVEN IN VOL 1, p A676; VOL 2, pp C215 to C216; VOL 3, pp XIV to XV; VOL 4, pp LI to LV AND VOL 5, pp XIV to XV

F.P. Bowden & A.D. Yoffe, "Fast Reactions in Solids", Academic Press, New York (1958), 164pp

R.E. Dupuy & T.N. Dupuy, "The Encyclopedia of Military History", Harper & Row, NY (1970) 1406pp

A.N. Dremin & co-workers, "Detonatsionnyiye Volny v Kondensirovannykh Sredadh (Detonic Waves in Condensed Media), "Nauka", Moscow (1970) (English translation in report-form, copied from 234 page transcript, is now available and awaiting a publisher at Per-Anders Persson Swedish Detonic Research Foundation, Vinterviken S-117 48 Stockholm Sweden)

E. Luttwak, "A Dictionary of Modern War", Harper & Row, NY (1971), 103pp

I.N. Bradley, "Flame and Combustion Phenomena", Halsted, NY (1972), 210pp

C.E. Gregory, "Explosives for North American Engineers", Trans Tech Publications, D-3392 Clausthal-Zellerfield, Adolf Ey Strasse 5, West Germany (1973), 288 pp

W.E. Baker, "Explosions in Air", Univ of Texas Press, Austin (1973), 268pp

E. Ya Orlova, "Chemistry and Technology of High Explosives" (Khimiya i Tekhnologiya Brizantnykh Vzryvchatykh Veshchestv), 2nd Ed, Khimiya, Leningrad, Otd, Leningrad, Russia (1973), 688pp

Sh.B. Bagdasarov, A.O. Vercheba, N.I. Kulichikhin, I.I. Pal'mov and A.D. Pashkov, "Handbook for a Mining Specialist in Geological Prospecting Groups with Special References to Properties of Explosives" (Spravochnik Gornogo Mastera Geologorozvedochnykh Partii), 2nd Ed, Nedra: Moscow, Russia (1973), 368pp

A.A. Ezra, "Principles and Practice of Explosive Metalworking", Industrial Newspapers Ltd, England (1973), 270pp

R.J. Pretty & D.H.R. Archer, Editors, "Jane's Weapon Systems", McGraw-Hill, NY, 5th edit (1973–74), 782pp

R. Liepens, R.W. Handy & J.W. Harrison, "Characteristics of Non-Military Explosives", Research Triangle Inst, Durham, NC (Feb 1974), 81pp (AD-778207/1WY) (Describes the compn of commercial expls and expl materials used for clandestine bomb fabrication)

C. Belgrano, "Gli Esplosivi", Arti Grafiche Friulane, Udine, 2nd edit (1974), 695pp

K.O. Brauer, "Handbook of Pyrotechnics", Chem Rubber Pubg Co, NY (1974), 412pp

E

Alphabet letter E begins in Vol 5, p E1

Etagenguss. German for *Multiple-Pouring or Step-Loading* method used during WWII for filling (Füllung in Ger) of large-caliber shells, such as of 15cm and 21cm. The method is described under "Loading of Ammunition"

Etching Reagent Explodes. Under this title Drs M.S. Frant & H.A. Fox Jr of AMP, Inc, Harrisburg, Pa stated (Ref 1) that 40ml of a well known etching reagent, prepd by them on mixing equal parts of acetone, concd nitric acid and 75% acetic acid, exploded about 4 hours after it was prepd and placed in a closed bottle

It was later suggested by W.J. Secunda of St Louis, Mo (Ref 2), that the formulation given in Ref 1 is very similar to that given for the prepn of TeNMe (Tetranitromethane). For its prepn, equal parts of acetic anhydride and concd nitric acid are allowed to stand for a few days. Since TeNMe is a good reagent for revealing a double bond (producing an intense yellow color with the same), it is suggested to test the etching soln of Ref 1 for the presence of TeNMe by bringing it in contact with an unsaturated compd

Refs: 1) C&EN, Oct 24, 1960, p 56-R 2) C&EN, Nov 14, 1960, p 5-L

Note: It might be of interest to know that TeNMe can be prepd by the action of hot fuming nitric acid plus concd sulfuric acid on Nitroform, as was done by Schischkoff in 1861 (Ann **119**, 247). The method of prepn of TeNMe from Ac_2O & HNO_3 was described by F.D. Chattaway, JCS **97**, 2100(1910). TeNMe is always present in small quantities in products of nitration of benzene, toluene, phenol, etc and can be recognized by a pungent odor

Ethanamide. See Acetamide in Vol 1 of Encycl, p A16-R

Ethanamidine, Ethaneamidine or **Ethenylamidine.** See α-Amino-α-imodoethane in Vol 1, p A223-L

Ethandiol-1,2. See Ethyleneglycol or Glycol

ETHANE AND DERIVATIVES

Ethane (Ethylhydride, Dimethyl, Methylmethane), $H_3C.CH_3$; mw 30.07; colorless & odorless gas, sl denser than air (1.04 vs 1.00 at 0° & 760mm); sp gr of liq 0.546 at −88°; fr p −183.23° (triple point); heat of combstn 22300 Btu/lb and expl limits in air (% by vol) lower 3.2 and upper 12.5. It is very sol in alc and moderately sol in water. It can be prepd by one of the following methods: fractionation from natural gas, petroleum or shale oil gases; also by passing alcohol vapor over amorphous Al at 360°

Its azide and nitro derivatives are explosive

Refs: 1) Beil **1**, 80, (22), [48] & {117} 2) CondChemDict (1961), 451-R & 452-L 3) Kirk & Othmer, 2nd edit **8**(1965), 383-85

Ethane Azide, Azidoethane, Ethyl Azide or **Triazoethane,** $H_3C.CH_2(N_3)$; mw 71.08, N 59.12%; liquid, sp gr 0.8765 at 25/4°; n_D 1.3928 at 25°; fr p not reported; bp 48.8–50° at 766mm; detonates at higher temp; stable at RT. It can be prepd by warming concd soln of Na azide with diethyl sulfate (Refs 1, 2 & 5). Campbell & Rice (Ref 3) investigated its phenomenon of expln and decided that it is apparently purely thermal, i.e. due to the accumulation of heat of reaction within the gas. The same scientists (Ref 4) investigated expln of Azidoethane in the presence of diethyl ether

Refs: 1) Beil **1**, [71] & {204} 2) H. Staudinger & E. Hauser, Helv **4**, 872(1921) 3) H.C. Campbell & O.K. Rice, JACS **57**, 1044–50 (1935) 4) O.K. Rice & H.C. Campbell, JChemPhys **7**, 700–09 (1939) 5) H.O. Spauschuss, JACS **73**, 208–09(1951)

Ethane, Azidochloro or **Chloroethyl Azide.** See under Chloroethane in Vol 3 of Encycl,, p C254

Ethane Azidonitrate, Ethylazido Nitrate or **Azidoethyl Nitrate,** $N_3.CH_2.CH_2.ONO_2$; mw 132, N 42.4%, OB to CO_2 −36.3%; toxic liquid, sp gr 1.34, fr p minus 20°, bp – decomp and expl at 180°, when heated at the rate of 25° per min. It is appreciably volatile. It is sol in benz, alc & eth; insol in water. It can be prepd by nitrating $N_3CH_2.CH_2.OH$ with mixed

acid (Refs 1 & 4)

Expl mixts are formed from Ethane Azidonitrate, NG, $NaNO_3$, wood meal & $CaCO_3$ or other mixts (Ref 2)

AzEtN gelatinizes NC and resembles in its physiological and explosive props NC and NG

Blatt (Ref 3), Urbański (Ref 4) and ADL (Ref 5) give the following props of AzEtN:
Impact Sensitivity: For a 50/50 mixt with sawdust, the max fall for no shots with a 10 lb wt was 2". The corresponding values for NGc and NG were 5" and 6", respectively. Absorbed on filter paper the pure liq could be detonated by the blow of a hammer
Power by Ballistic Mortar – 141% TNT
Power by Trauzl Test – 150% TNT
Velocity of Detonation – 1985 and 6550m/sec at sp gr 1.34
Refs: 1) Beil 1, {1365} 2) F.H. Bergeim, USP 1620714 & 1620715(1927) & CA **21**, 1551(1927) 3) Blatt, OSRD **2014**(1944) (Azido) 4) T. Urbański, IX CongrIntem-QuimPuraApplicata **4**, 438(1934) & CA **30**, 3649(1936) 5) ADL, PureExplCompds, Part 1(1947), pp 48 & 150

Ethane Diazide; Diazidoethane, Ethyldiazide or **Bistriazoethane,** $H_3C.CH(N_3)_2$; mw 112.10, N 74.98%, OB to CO_2 –85.6%

The following isomers exist:

Ethane-1,1-Diazido or **1,1-Diazidoethane (1,1-DAzEt)**, solid, extremely unstable, even at RT. The first attempt to prep it in 1908 (Ref 2) failed because a disastrous expln occurred and its prepn was not repeated
Refs: 1) Beil – not found 2) M. Forster et al, JCS **93**, 1073(1908)

Ethane-1,2-Diazide or **1,2-Diazidoethane (1,2-DAzEt)** or **1,2-Bistriazoethane,** $N_3.CH_2.CH_2.N_3$; oil with chlf-like odor, sp gr 1.178 at 19/19°, fr p – not reported, bp 53° at 9mm; explodes when thrown on a hot plate. It can be prepd by heating ethanol, ethyl chloride and Na azide in water

It is an expl nearly as powerful as TNT (97% by Trauzl Test) and moderately sensitive to friction and impact
Refs: 1) Beil 1, 103 & {204} 2) M.O. Forster et al, JCS **93**, 1071(1908) 3) Blatt, OSRD **2014**(1944) 4) ADL, PureExplCompds, Part 1(1947), p 44

ETHANE NITRATED AND NITROSATED DERIVATIVES

Ethane Nitrate, Ethyl Nitrate or **Ethane Nitric Ester** (Äthyl Nitrat or Salpetersäure-äthylester, in Ger), $H_3C.CH_2.ONO_2$; mw 91.07, N 15.38%, OB to CO_2 –61.5%; colorless, pleasant smelling, volatile liquid which can be easily ignited by flame; sp gr 1.044 at 25/25°, or 1.116 at 15°, n_D 1.387, fr p –102°(–112°); bp 87.5°, fl p 34°F; vapor density 3.14(Air 1.0). It is sl sol in w (1.3% at 53°) and is miscible with alc or ether; gelatinizes collodion cotton. It was first prepd by Millon in 1843 by treating absol alc with nitric acid (d 1.4) in the presence of urea which served to eliminate lower N oxides (Ref 1, p 329 & Ref 1a). A modified Millon method is described by Naoúm (Ref 2a). The highest yield obtained by method of Millon and its modifications was only 35%. Ville (Ref 6) describes in detail the procedure which is claimed to give a yield of 85% (calcd on consumed alc)
Procedure. Introduce into a 3-neck, round-bottom flask of 350ml capacity (provided with an agitator and an ice-water cooling bath) 80g of mixed acid of approx compn: HNO_3 53, H_2SO_4 42 & SO_3 5%. The acid is obtd by mixing, with stirring and cooling, calcd quantities of 95% strong HNO_3 (freed from lower N oxides either by urea or by bubbling thru dry air at 60°) and ca 20% strong oleum. When the temp of mixed acid reaches ca 10°, add drop by drop a mixt of 26g alc (95% strong) & ca 2.5g urea, while stirring and maintaining the temp at 10–15°. Transfer the liquid to a separatory funnel, stopper it and shake gently. Remove thru the stopcock the lower (acid) layer and discard it. Wash the liquid remaining in the funnel, first with 20ml of iced w and then with 20ml of cold 2% Na carbonate soln. For drying and eliminating residual acid, add few grams of anhyd Na carbonate and transfer EtN into weighing bottle. By using this method, Ville obtd 185g of product of d 1.102, which was 96–97% pure. When ignited, the product burns quietly with clear flame, but Ville could not explode it by impact or by a detonator contg 1g MF (Ref 6, pp 79–80)

Naoúm (Ref 2a) gives the following props: Notwithstanding its high negative oxygen balance, it is a powerful and brisant expl. With decompn accdg to the equation:

$2C_2H_5.ONO_2 = 4CO + 2H_2O + N_2$,

the heat of expln may be calcd, if the heat of formation is taken as 48.5kcal/mole, as given in Ref 2, p 207. The heat of expln arrived at in this manner is 712.6kcal/kg at C_v and H_2O gas or 816.3kcal/kg with H_2O liquid. These values are about 50% of those for NG (1485 and 1585kcal/kg)

Schmidt (Ref 3) gives a different value for its heat of formation, namely 33.7kcal/mole or 370kcal/kg (vs 82.7kcal/mole & 364kcal/kg for NG). He also gives heats of combustion at C_v, H_2O liquid (3561kcal/kg for EtN & 1631 for NG), but no heats of expln

Blatt (Ref 4b) gives its heat of combstn at const vol as 324kcal/mole. Hutchison (Ref 5a) gives its CVWL (calorific value, water liquid) as 850cal/g

Blatt (Ref 4b) gives its power by Ballistic Mortar Test as 123% TNT, while by Trauzl Test 345cc for 10g sample initiated by No 8 blasting cap and under water tamping. This value is about 58% that of NG (590cc). EtN is rather insensitive to detonation and is less sensitive to shock or friction than NG or NGc. It can be detonated with No 8 cap when confined or when mixed with some powdery material, such as kieselguhr. In the latter case it can be initiated without confinement. Its detonation velocity was detd indirectly in mixts consisting of EtN 60 to 70 & kieselguhr 40 to 30% and was found to be of the magnitude of 6000 to 7000m/sec (Refs 2a & 4a)

It is stable in storage at ordinary temps

In Ref 4 are given its flash point as 50°F (in open or closed cup) and its lower explosive limit in mixts with air as 3.8% by vol. No upper limit is given. In Ref 5 is discussed its detonation by impact and in Refs 7 & 8 are briefly discussed its toxicity and fire & expln hazards. Hutchison (Ref 5a) gives its specific impulse (theoretical) as 197, viscosity 0.6c/stokes at 20° and temp of decompn 1600°. It will only propagate detonation in a pipe of diam greater than 1¼ inches

Accdg to Midgley & Boyd (Ref 2), EtN added in a small quantity to a liquid fuel used in internal combstn engines accelerates the combstn

Uses: Medecine, organic synthesis, commercial expls and liquid rocket proplnts (Refs 2, 2a, 5a, 7 & 8)

Hutchison (Ref 5a) discusses in detail the use of EtN in liq rocket proplnts. The use of EtN is not recommended as a means of operating aero engine starters, or for any other operations where the unit is to be repeatedly used in close proximity to human life

It was recommended in France as a dope for fuels intended for use in two-cycle engines (moteurs à deux temps)

Refs: 1) Beil **1**, 329, (165), [328–29] & {1322} 1a) N. Millon, Ann **47**, 373(1843) 2) T. Midgley & T.A. Boyd, IEC **14**, 896(1922) 2a) Naoúm, NG(1928), 206–08 3) A. Schmidt SS **29**, 262(1934) 4) Associated Factory Mutual Fire Insurance Company, IEC **32**, 882 (1940) 4a) Davis (1943), 194–95 4b) Blatt, OSRD **2014**(1944) (Ethyl Nitrate) 5) F.P. Bowden et al, ProcRoySoc **A188**, 306–07 5a) A.C. Hutchison, "The Use of Alkyl Nitrates as Liquid Monofuels", ICI (Imperial-ChemicalInd) Ltd, NobelDivision, Stevenston, Ayshire(1950) 6) J. Ville, MP **35**, 71–82 (1953) & CA **49**, 6082(1955) 7) Sax (1957), 698 8) CondChemDict (1961), 474 9) Kirk & Othmer, 2nd edit, not found

Addnl Refs: A) N.V. Sidgwick, "Organic Chemistry of Nitrogen", OxfordUnivPress, London (1937), pp 8 & 250 [EtN may be used for some difficult nitrations, as for example to 1,3,6,8-Tetranitrocarbazole in presence of sulfuric acid. It has been chiefly used, however, for replacing a hydrogen in a reactive methylene group by NO under the influence of Na ethoxide:

$R.CH_2.COOC_2H_5 + C_2H_5.ONO_2 + KOC_2H_5 \rightarrow$
$R.C(:NO.OK).COOC_2H_5 + 2C_2H_5OH$

A weaker action is obtd when EtN reacts in presence of $AlCl_3$, as for example benzene or toluene are nitrated to mono- stage]

B) A.C. Egerton & J. Powling, ProcRoySoc (London) **193A**, 172(1948) & CA **43**, 3617(1949) (Explosibility of EtN)

C) P. Gray & A.D. Yoffe, JChemSoc **1950**, 3180 & CA **46**, 25–6(1952) (Flammability of EtN)

D) J.B. Levy, "The Effect of Additives on the Thermal Decomposition of Ethyl Nitrate", NAVORD Rept **2897**(1953)

E) L. Médard, MP **36**, 75(1954) & CA **49**, 16435(1955) (Expl props of EtN)
F) R. Vandoni & M. Laudy, MémServicesChim-État **40**, 187(1955) (Vapor pressure in mm: 9.1 at −10°, 16.5 at 0° and 30.5 at 10°)
G) E.A. Blyumberg & V.L. Pikaeva, ZhurAnal-Khim **10**, 310(1955) & CA **50**, 2369(1956) (Polarographic determination)
H) G. Desseigne & R. Robussier, MP **39**, 172–79(1957) (Prepn of EtN)
I) G. Desseigne, FrP 1164368(1958) & CA **53**, 461(1959) (Continuous method of prepn)

Ethane Nitrite, Ethyl Nitrite, Ethane Nitrous Ether or **Nitrosoxyethane** (Äthyl Nitrit or Salpetrigsäure-äthylester, in Ger), $H_3C.CH_2.ONO$; mw 75.07, N 18.66%; lt yel, volatile & flammable liq or gas; sp gr 0.900 at 15.5°, vapor density 2.59 (Air 1.0), fr p – not given, bp 17°; v sl sol in w, miscible with alc or eth. It was first prepd in 1681 by Kunkel in impure state by treating ethanol with nitrous acid (Ref 1). Wallach & Otto (Ref 2) prepd it by treating ethanol with Na nitrite in presence of sulfuric acid. It was also prepd by Strecker & Spitaler (Ref 3). Adickes (Ref 4) describes a special apparatus in which is automatically achieved complete mixing of aq alcohol–Na nitrite and sulfuric acid in order to avoid the sudden dangerous evolution of gas during nitrification. Detailed description of prepn is given in OrgSynth (Ref 5). Two methods of prepn are listed in CondChemDict (Ref 8): a) By the action of nitrous oxide gas on ethanol; and b) By treating ethanol with alkali nitrites and sulfuric acid

In Ref 4 is given its fl p as 30°F in open cup & 31°F in closed cup and its expl limits in mixts with air 3.1 to >50% by vol. Toxicity and fire & expln hazards are discussed in Refs 7 & 8. Its explosibility is discussed in Ref 6

Ethyl Nitrite has been used in medicine under the name "*spiritus aetheris nitrosi*"
Refs: 1) Beil **1**, 329, (165), [328] & {1321} 2) O. Wallach & A. Otto, Ann **253**, 251(1889) 3) W. Strecker & R. Spitaler, Ber **59**, 1774(1926) 4) Associated Factory Mutual Insurance Companies, IEC **32**, 882(1940) 4) F. Adickes, JPraktChem **161**, 275(1943) & CA **38**, 65(1944) 5) "Organic Syntheses", Collective Vol **2** (1943), p 204 6) A.C. Egerton & J. Powling, ProcRoySoc(London) **193A**, 172(1948) & CA **43**, 3617(1949) (Explosibility of Et Nitrite) 7) Sax (1957), 698 8) CondChemDict (1961), 475 9) Kirk & Othmer, 2nd edit, not found

Nitrosoethane (NsoEt) or **Nitrosoethyl**, $CH_3.CH_2NO$; mw 59.07, N 23.71%; wh waxy solid having a sweet penetrating odor, mp 58–62° to a blue liq which became pale green as the temp rose; the liq charred at 138°. It was formed, with other products, when di-n-butylmercury, Bu_2Hg, was pyrolized in the presence of NO as carrier gas in the temp range 376–410° (Ref 2). It was also formed at temps above 100° by the addn of ethane to NO_2 (Ref 3). Its prepn by photolyzing ethyl iodide in the presence of NO and the kinetics of the reaction are reported by Christie et al (Ref 3)
Refs: 1) Beil, not found 2) H.T.J. Chilton & B.G. Gowenlock, JChemSoc **1954**, 3175 & CA **49**, 6822(1955) 3) T.M. Rohr & W.A. Noyes, CanJChem **33**, 843(1955) & CA **49**, 12971(1955) 4) M.I. Christie et al, TransFradSoc **61**(508), 674(1965) & CA **62**, 16014 (1965)

Nitroethane or **Nitroethyl (NEt)** (Nitroäthan, in Ger), $H_3C.CH_2.NO_2$; mw 75.07, N 18.66%, OB to CO_2 −95.9%; colorless, flammable liq; sp gr 1.052 at 20/20°; fr p −90°, bp 114° at 760mm, vapor pressure 15.6mm, flash point (Tag open cup) 106°F, n_D 1.3917 at 20°; heat of combstn 4299cal/g, heat of formation 472cal/g; sl sol in w; v sol in alc, eth or chlf; sol in acids and alkalies

It was first prepd in 1894 by Nef (Refs 1 & 2) by nitrating ethane. Accdg to CondChemDict, NEt can be prepd by treating ethane with oxides of nitrogen or with nitric acid under pressure (Ref 9). In the method of prepn listed by Fieser & Fieser (Ref 7), vapors of ethane pass together with vapors of nitric acid thru a narrow reaction tube at 420°. Accdg to Hibshman et al (Ref 4), vapor-phase nitration of ethane produces a mixture of C_2H_5NO with CH_3NO_2. In the lab method of prepn by Desseigne & Giral (Ref 8), pure NEt is produced starting from ethanol, oleum and Na nitrate. It is stated, however, that the method is not suitable for industrial purposes because it cannot compete with vapor-phase nitration of propane which renders

considerable proportion of NEt. In the lab method of McCombie et al (Ref 6), NEt was prepd by the action of Na nitrite on ethyl sulfate: $C_2H_5O.SO_2.OC_2H_5 + NaNO_2 \rightarrow C_2H_5O.SO_2ONa + C_2H_5NO_2$

Accdg to McKittrie et al (Ref 3), NEt explodes at 450° after few minutes of heating. Blatt & Whitmore (Ref 5) found it difficult to detonate

It can be used as a solvent for NC in prepn of propellants

Salts of NEt

Sodium and potassium salts were first prepd and examined by Nef (Ref 2). For their prepn NEt was dissolved in ether and treated with Na or K alcoholates (obtd by dissolving metallic Na or K in alcohol). Both $C_2H_4(NO_2)Na$ and $C_2H_4(NO_2)K$ are sol in w and very sol in alc. The Na salt deflgr on heating & explodes at high temp. The Thalium salt, $TlC_2H_4NO_2$, crysts (from aq alc), dec at 80–82° (Ref 1)

Refs: 1) Beil **1**, 99, (32), [70] & {199} 2) J.U. Nef, Ann **280**, 273(1894) 3) D.C. McKittrie et al, IEC(AnalEd) **10**, 63(1938) 4) H.B. Hibshman et al, IEC **32**, 427–29 (1940) 5) A.H. Blatt & F.C. Whitmore, OSRD **1085**(1942), 63 6) H. McCombie et al, JCS **1944**, 24–5 7) L. Fieser & M. Fieser, "Organic Chemistry", Heath & Co, Boston (1950), 48 8) G. Desseigne & H. Giral, MP **34**, 49–53(1952) 9) CondChem-Dict (1961), 803-R & 804-L

Dinitroethanes (DNEt), $C_2H_4(NO_2)_2$; mw 120.07, N 23.33%, OB to CO_2 −26.7%

The following isomers exist:

1,1-Dinitroethane (1,1-DNEt), $(O_2N)_2CH.CH_3$; colorless oil with faint alcoholic odor; fr p – not reported; bp 185–86° at 760mm and 73–74.5° at 13mm, n_D 1.4340 at 20°; sp gr 1.3503 at 23.5°

It can be prepd by acidification of potassium-1,1-dinitroethanate, first prepd in 1876 by Ter Meer (Refs 1 & 2). In A.D. Little Inc Rept (Ref 6, pp 81, 107 & 108), the prepn of the Potassium salt is described using Ter Meer's method, as modified by Jacobson (Ref 4). For this, to 1-chloro-1-nitroethane (listed in Vol 3 of our Encycl, p C254-R) dissolved in methanol, was added at about 0° an aq soln of K nitrite. A short time after the start of addition (temp reached about 5° but below 10°) a stream of methanolic KOH soln was started to be added. The addn of the two solns was at such a rate that addn of KNO_2 was completed just before that of the KOH soln. The salt was collected on a filter funnel and washed with ice water & then cold methanol. The crude product was recrystd from water

Bright yel crysts of $KC_2H_3(NO_2)_2$ obtd exploded at 150°. The same salt can be obtd on treating 1-bromo-1-nitroethane (Vol 2 of Encycl, p B311-R) with K nitrite in alcoholic soln of KOH (Ref 3). It explodes violently by impact

For prepn of 1,1-DNEt, ADL Rept (Ref 6, p 108) recommends acidifying the K salt by means of 85% phosphoric acid and purifying the resulting product by vacuum distillation **behind a barricade.** A small amt of boric acid was added before the distillation operation. The water-white distillate boiled at 73–74.5° at 13mm pressure

Blatt (Ref 5) lists the following props of 1,1-DNEt:

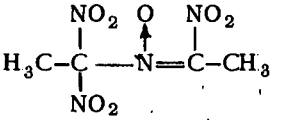
Power, by Ballistic Mortar Test – 138% TNT

Sensitivity to Impact – comparable with PETN

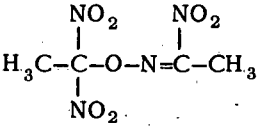
Stability at RT – decomposes with evolution of nitrogen oxides on standing for several weeks

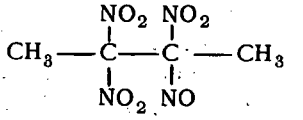
Thermal Stability at 135° – acidic in 30 min and no expln in 300 mins

Uses: In ADL Rept (Ref 6, p 108) is described the use of 1,1-DNEt or its K salt in prepn of so-called **Compound 335,** which is of unknown structure but considered by some investigators to be 2,2,3,3-Tetranitrobutane:



or





Reactions of 1,1-DNEt with its salts are described in Ref 7. The *Ammonium* salt, $NH_3C_2H_3(NO_2)_2$, yel salt, mp 90–93° (dec) was prepd and so were others of 1,1-DNEt

Refs: 1) Beil **1**, 102, (32) & [70] 2) E.

Ter Meer, Ann **181**, 1(1876) 3) H. Wieland & E. Sakellarios, Ber **52**, 904(1919) 4) H.W. Jacobson, "PhD Thesis", Purdue University (1942) 5) A.H. Blatt, OSRD **2014**(1944) 6) ADL, Synthesis HE's, 2nd Rept (1951), pp 81 & 107–08 7) J.S. Belew et al, JACS **77**, 1113(1955) & CA **50**, 1648 (1956) 8) K. Torsell & R. Ryhage, ArkivKemi **23**(44), 525(1965)(Engl) & CA **63**, 6839(1965) (Expln of K salt at 150°)

1,2-Dinitroethane, $(O_2N)CH_2.CH_2(NO_2)$; wh crysts (from MeOH or benz), mp 39–40°, bp 135° at 5mm press; slowly decomp on storage; insol in w (Refs 1 & 4); can be prepd in 70–80% yields by passing ethylene, C_2H_2:C_2H_2, into nitrogen tetroxide, N_2O_4, at −10°. The yield reported in Blatt (Ref 3) was only 20–30%

It reacts with urea forming Nitroethylene:

$$2\begin{vmatrix} CH_2.NO_2 \\ CH_2.NO_2 \end{vmatrix} + CO(NH_2)_2 = \begin{matrix} CH_2 \\ \| \\ CH.NO_2 \end{matrix} + 2N_2 + CO_2 + 3H_2C$$

When heated with PETN at 100°, 1,2-DNEt reacts very violently

The following props are given in Blatt(Ref 3):
Friction Sensitivity – very insensitive
Impact Sensitivity – less sensitive than PA
Initiation Sensitivity – very insensitive when cast
Power, by Trauzl Test – 146% PA
Power, by Ballistic Mortar – 91% of Blasting Gelatin
Stability – unstable turning acidic on standing

1,2-DNEt was also prepd and examined during WWI by Ipatow (Ref 2). See Ref 4 which questions the identity of product obtd as grn-yel oil, bp 94–96° at 5mm press
Refs: 1) Beil **1**, (32) & {203} 2) A.V. Ipatow, JRussPhysChemSoc **49**, 300(1917) & CA **17**, 3158(1923) 3) A.H. Blatt, OSRD **2014**(1944) 4) N. Levy et al, JChemSoc **1946**, 1098 5) L. Médard & M. Thomas, MP **36**, 99(1954) & CA **50**, 3763(1956) (Detd Heat of combstn as 283.9 & Heat of formation as 40.1kcal/mole, both at const vol & at 18°)

Trinitroethanes (TNEt), $C_2H_3(NO_2)_3$; mw 165.07, N 25.46%, OB to CO_2 +8.45%

The following isomers exist:

1,1,1-Trinitroethane, $(O_2N)_3C.CH_3$, small white, very volatile cubic crysts; sp gr 1.4223 at 77.7°, mp 56–57°, bp 68° at 17mm press; explodes at higher temp; v sl sol in w and in cold ligroin; sol in alc, eth and in most org solvents; heat of combstn at C_v 1777cal/g. It detonates by a hammer blow; can be prepd by the action of a cooled ethereal soln of silver nitroform on an excess of methyl iodide: $C(NO_2)_3Ag + CH_3I = C(NO_2)_3.CH_3 + AgI$. After allowing the mixture to stand for a short time, it is filtered to remove AgI and the filtrate evaporated in vacuum. In the method of prepn described in US Rubber Repts (Ref 4), K nitroform was dissolved in acetone and treated with methyl iodide; mp of TNEt was reported as 53–4°, while Hantzsch & Rinckenberger (Ref 2) gave 56°

In US Rubber Rept No 25 (Ref 6) is described the method according to Rohm & Haas Co QuartRept No **P52–3**, Sept 1952, p 12. See Refs for addnl methods of prepn and for other properties
Refs: 1) Beil **1**, 103, (33), [70] & {204} 2) A. Hantzsch & A. Rinckenberger, Ber **32**, 636(1899) 3) K. vonAuwers & L. Harries, Ber **62**, 2296(1929) 4) US Rubber Rept **No 4**(1948), 9–10; **No 7**(1948), 5–6; **No 8** (1948), 25; and **No 25**(Nov 1953–Feb 1954) (Contracts NOrd 10129 & 12663) 5) H. Wittek, ActaPhysAustriaca **1**, 303(1948) & CA **42**, 6665(1948) (Raman spectrum) 6) A. Audsley & F.R. Goss, JChemSoc **1949**, Suppl No 1, S228 & CA **44**, 392(1950) (Dipole moment) 7) L. Zeldin & H. Shechter, JACS **79**, 4708(1957) & CA **52**, 2734(1958) (Prepn & reactions with org bases) 8) Ya.K. Syrkin et al, DoklAkadNauk **132**, 1376(1960) & CA **55**, 15036(1961) (Dipole moment) 9) V.I. Solvetskii et al, IzvestAkadNauk, SSSR, OtdelKhimNauk **1961**, 330 & CA **55**, 19473 (1961)(IR & UV spectra) 10) C.W. Plummer, USP 3049570(1962) & CA **57**, 15404(1962) (Prepn as expl compd) 11) G.S. Hammond et al, Tetrahedron **19**, Suppl 1, 190(1963) & CA **59**, 11237(1963) (Prepn from CH_3I & Ag nitroform in acetonitrile) 12) A.P. Hardt et al, ElectrochemTechnol **1**, 375(1963) (Engl) & CA **64**, 3047(1966) [Prepn from 1,1-Dinitroethane (or Nitroethane) thru anodic nitration in aq 10N NaOH–10N $NaNO_2$ soln below RT] 13) M. Masui & H. Sayo, ChemPharmBull(Tokyo) **14**(3), 306(1966) (Engl) & CA **65**, 1640(1966) (Electron spin resonance spectrum)

1,1,2-Trinitroethane, $(O_2N)_2CH.CH_2(NO_2)$.

This compd is listed in Table 139 of Ref 2, as compound 569. No refs as to its prepn & props were found in the literature
Refs: 1) Beil – not found 2) ADL Pure-ExplCompds, Part 4 (1952), p 570

Tetranitroethanes (TeNEt), $C_2H_2(NO_2)_4$, mw 210.07, N 26.67%, OB to CO_2 +22.8%

The following isomers are theoretically possible:
1,1,2,2(or sym)-Tetranitroethane, $(O_2N)_2CH.CH(NO_2)_2$. This compd could not be prepd but is known in the form of its salts, such as:

Dipotassium-1,1,2,2-Tetranitroethanate (DKTeNEt), $K_2C_2(NO_2)_4$; mw 286.25, N 19.57%, OB to CO_2 & K_2O +16.8%; small, glittering yel crysts with violet tinge (from aq methanol); mp – explodes at 267–275°; v sol in w; sol in aq methanol; sl sol in methanol, alc, acet, benz, chlf & AcOH. It was first prepd by Scholl & Brenneisen (Refs 1 & 2). Further work was done by Scholl & Schmidt (Ref 3) and Will (Ref 4). The yields were very poor because many side reactions took place, in addition to the following main reaction:

$2C(NO_2)Br_3 + KCN = KBr + BrCN +$
Bromopicrin Cyanogen bromide

$C(NO_2)Br_2.C(NO_2)Br_2$
Tetrabromodinitroethane

$C(NO_2)Br_2.C(NO_2)Br_2 + 2KNO_2 = 2KBr +$
$C(NO_2)_2Br.C(NO_2)_2Br$
Dibromotetranitroethane

$C(NO_2)_2Br.C(NO_2)_2Br + 2KCN = 2BrCN +$
$C(NO_2)_2K.C(NO_2)_2K$
Dipotassium Tetranitroethane
(or $2KNO_2$) = $2BrNO_2$ (Nitryl bromide)

Due to the fact that the salt is extremely sensitive, especially when in an impure state, it is advisable to prepare it in smaller quantities than was suggested by Will (Ref 4)
Laboratory Procedure:
(Work should be done behind a protecting screen of safety glass or plastic). Dissolve about 50g of bromopicrin in about 25ml of methanol (contained in a 400ml beaker immersed in a bath with tap water) and add slowly, with constant stirring and cooling, 25g of finely powdered potassium cyanide, followed by a solution of 12.5g of potassium nitrite in about 40ml of water. Do not allow the temperature of the mixture to rise above 30°. Stir for an additional 1/2 hour, cool in an ice-salt bath (to get better yield) and filter the pptd salt thru a Büchner funnel. Dissolve the precipitate in the smallest amount of warm water containing a small amount of KOH (to render alkaline), and crystallize by freezing the solution in an ice-salt bath. Remove the crystals by filtration and dry them at 80–90°. Yield about 8.5g. Keep the crystals in a beaker wrapped on the outside with a piece of cheesecloth (this is recommended because the salt is very sensitive and might accidently explode)

Other methods of preparation are described by Hunter (Refs 5 & 6) and by Houben (Ref 8)

DKTeNEt is an explosive of the initiating class and is extremely sensitive to friction, impact and heat, especially if not properly purified

The salt was prepd at Picatinny Arsenal in 1930 by S. Livingston (Ref 7) and then in 1947 by Dr K.S. Warren. However, the compound was considered too dangerous for use as a military explosive and work on it discontinued after what was supposed to be some of the salt exploded in a beaker resulting in the death of one chemist, Dr P.F. Macy

Silver Tetranitroethanate, $Ag_2C_2O_8N_4$. Yellow-red crystalline compound, insol in w, alc & ether; was first prepd by Scholl and Brenneisen (Ref 2, p 649) by adding a concd aq solution of 1.7g of $AgNO_3$ to a cold concd aq solution of 1.5g of dipotassium tetranitroethanate. After allowing to stand for a while, the salt crystallized out and was obtd by filtering the mixture thru a small Büchner and washing with alcohol and ether

It is a very mild explosive practically insensitive to impact

Lead Tetranitroethanate, $PbC_2O_8N_4$. Brown crystalline compound, claimed to have been prepd by S. Livingston (Ref 7) by dissolving 1g of the potassium salt in about 25ml of water and adding to this about 15ml of water containing 1.3g of lead acetate. The precipitated lead salt was filtered, washed with w, alcohol & ether. It was insol in w, alc and eth and was a mild expl, practically insensitive to impact. When brought into contact with a flame, it burned rapidly without expl

Refs: 1) Beil **1**, 103, (33), [70] & { 204} 2) R. Scholl & M. Brenneisen, Ber **31**, 646–47 (1898) 3) R. Scholl & A. Schmidt, Ber **35**, 4288(1902) 4) W. Will, Ber **47**, 963–64(1914) 5) L. Hunter, JCS **123**, 547(1923) 6) L. Hunter, JCS **125**, 1484(1924) 7) S. Livingston, PicArsnTechRept No **R85**(1930) 8) J. Houben, "Die Methoden der Organischen Chemie, G. Thieme, Leipzig, Vol **4**(1941), p 179 9) F.G. Borgardt et al, JOrgChem **31**(9), 2806 (1966) & CA **65**, 13523(1966) (Prepn of Dipotassium salt from 1,1,1-trinitrochloroethane by treatment with nitrite ion in basic media)

1,1,1,2-Tetranitroethane, $(O_2N)_3C.CH_2.NO_2$. No information at our disposal that it was ever prepd; not found in the literature thru 1966

1,1,1,2,2-Pentanitroethane, $(O_2N)_3C.CH(NO_2)_2$. No information at our disposal that it was ever prepd; not found in the literature thru 1966

HEXANITROETHANE (HNEt) (Called Hexanitroäthan, in Ger), $(O_2N)_3C.C(NO_2)_3$; mw 300.07, N 28.01%, OB to CO_2 42.7%; colorless crysts (from ether), nonhygroscopic, sl volatile at RT, mp 142° (with sublimation and partial decompn); explodes mildly on rapid heating above 360° and by action of a detonator. Soluble in ether, benz, petr eth & chlf; diffc sol in ethanol; insol in water

It was prepd in 90% yield by Will (Refs 1 & 3), but in 1913 it was patented by Claessen for use as an ingredient of expls (Ref 2 and Vol 3 of Encycl, p C325-R). Will (Ref 3) prepd HNEt in 1914 by nitrating a concd sulfuric acid soln of pure Dipotassium Tetranitroethanate (qv) with mixed nitric-sulfuric acid at 5 to 10°C. Prepn of dipotassium salt from bromopicrin and potassium cyanide is described above under Tetranitroethane. In the method described by Houben (Ref 7), a 100g sample of dipotassium salt is used. As such a big sample is too dangerous for laboratory method, we suggest reducing it to 10g and proceeding as follows (while working behind a safety screen, under a hood): Dissolve a 10g sample of very pure DKTeNEt in 50ml of concd sulfuric acid, with constant stirring and cooling to 5°C. Then add slowly the mixed acid (consisting of 15ml nitric acid of sp gr 1.52 and 15ml concd sulfuric acid), with stirring and cooling. Raise gradually the temp to 60–70° and maintain it for 10 mins. Then cool in ice bath and drown immediately in a large volume of ice-water. Filter by suction thru a sintered glass Büchner funnel, wash crysts of HNEt with cold w, dry them by passing air and dissolve in ether. Add some powdered $CaCO_3$ to neutralize the acidity, filter and evaporate the ether by air. The yield was claimed by Houben to be 90% of theory

Note: If Dipotassium Tetranitroethane is not pure, it would ignite immediately on contact with concd sulfuric acid

HNEt was also prepd at the NatlFireworks (Ref 10) and the US Rubber Co Lab (Ref 11). Noble et al (Ref 15) reported some of its physical and chemical properties:

Explosive Properties of HNEt [as listed by Will (Ref 3), McGill (Ref 7a), Blatt (Ref 8) and ADL (Ref 9)]

Impact Sensitivity – comparable with PETN(FI)

Power by Ballistic Mortar Test – 108% TNT

Power by Trauzl Test – 115% TNT

Thermal Stability at 135° – acidic in 30 mins

Uses: Due to the presence of excess O, HNEt forms in mixts with HE's deficient in O, very powerful expls. Claessen (Ref 2) patented in 1913 several mixts with TNT suitable as bursting or booster chges of artillery shells. Some of them, such as HNEt 45 & TNT 55% are listed in Vol 3 of Encycl, p C325-R. He also patented its use in manuf of artillery proplnts, such as: NC 68, HNEt 16, TNT 9 & Centr 1%, listed in Vol 3, p C326-R. A blasting expl contg HNEt 64 & TNT 36% was claimed to be more powerful than Blasting Gelatin. These expls are also listed in Colver (Ref 5). During WWI HNEt was used in "Big Bertha" ammunition. Big Bertha, also known as "Paris Gun" (Parisgeschüt or Pariserin, in Ger) is briefly described in Vol 2 of Encycl, p B113-R

Hannum (Ref 12) patented mixts of fuels, consisting of NMe (Nitromethane), NEt (Nitroethane), and mono-, di-, or trinitropropane, with oxidants HNEt or TeNMe (Tetranitromethane), stabilized by the addn of substituted ammonias in amts of 1 to 10% by wt. A preferred mixt is NMe 55.5 & HNEt (or TeNMe) 44.5%, with p-phenylenediamine or Centralite as a stabilizer

Hannum also patented (Ref 13) an expl

mixt of paraffin 10–40 and TeNMe or HNEt 60–90%, which was claimed to be sensitive to expl shock but resistant to impact
Refs: 1) Beil **1**, (33) & [71] 1a) Zentralstelle für Wissenschaftlich-Technische Untersuchungen GmbH, GerP 281906(1913) (Prepn of HNEt) 2) C. Claessen, FrP 463714(1913) & CA **8**, 3238(1914); BritP 24839(1913) & CA **9**, 1249(1915) 3) W. Will, Ber **47**, 961 (1914) & CA **8**, 1784(1914) 4) Vereinigte Köln-RottweilerPulverfabrik, GerP 277594 (1915) & CA **9**, 862(1915) (Properties and mixts with NC) 5) Colver (1918), 396–98 & 725–26 (Expl mixts with HNEt) 6) Stettbacher (1933), 205 (Proplnts contg HNEt) 7) J. Houben, "Die Methoden der Organischen Chemie", G. Thieme, Leipzig, Vol **4**(1941), p 171 (Prepn of HNEt from Dipotassium Tetranitroethanate) 7a) R. McGill, OSRD **830** (1942) (Prepn & props of HNEt) 8) A.H. Blatt, OSRD **2014**(1944) (Some expl props) 9) ADL, PureExplCompds, Part 1(1947), p41 (Some expl props of HNEt) 10) National-Fireworks,Inc, Summary Rept, Dec 1948 to Aug 1949, Contract NOrd 10111 (Prepn of HNEt) 11) USRubber Co, Rept **No 8**(1949), p 10 (Prepn of HNEt) 12) J.A. Hannum, USP 2442193(1951) & CA **45**, 10545–46(1951) (Expl mixts contg HNEt) 13) J.A. Hannum, USP 2560439(1951) & CA **46**, 739–40(1952) (Expl mixts contg HNEt) 14) B.E. Holder & M.P. Klein, JChemPhys **23**, 1956(1955) & CA **50**, 2285(1956) (Nuclear magnetic resonance) 15) P. Noble, Jr et al, AIAA (American Institute of Aeronautics & Astronautics) **1**, 395–97(1963) (Props of HNEt)

Ethanemercarbide and Derivatives

Ethanemercarbide (called Dianhydro-hexakis-[hydroxymercuri]-äthan, or Äthanmercarbid, in Ger), $HO.Hg.(OHg_2)C.C(Hg_2O).HgOH$; mw 1293.69, OB to CO_2 –8.7%, yel powd, detonates at 230°C (183° C). It may be prepd by heating EtOH, PrOH, allyl-OH, amyl-OH, AcH, cane sugar, starch, or cellulose w yel HgO in alk soln; insensitive to impact, but crackles under friction. It dehydrates at 200–230°C prior to violent detn at 230°C; not decompd by strong acid or alkali (Ref 2b)

Ethanemercarbide Dinitrate, $O_2N.O.Hg.(HO.Hg)_2C.C(HgOH)_2.HgONO_2$; mw 1419.73, OB to CO_2 –2.3, yel-wh crysts; decomp on heating. It may be prepd by treating the parent compd with 10% nitric acid; insol in water & dil nitric acid (Ref 2a)

Ethanemercarbide Dinitrate Diammoniate, $C_2H_6.Hg_6N_4O_8$; mw 1417.76, OB to CO_2 –5.6%; may be prepd by treating the dinitrate with an AN-NH_4OH soln for 6 hrs at 30°C (Ref 2b)

Ethanemercarbide Disulfate, $HO_3SO.Hg.(HO.Hg)_2C.C(HgOH)_2.Hg.O.SO_3H$; mw 1489.84, OB to CO_2 –7.5%, wh cryst powd; insol in w, decomp on heating; may be prepd by boiling the dinitrate with NaOH soln, then heating with 20% sulfuric acid (Ref 2a)

Dithiaethanemercarbide, $C_2H_2Hg_6O_2S_2$; mw 1325.82, OB to CO_2 –18%, yel powd. It may be prepd by treating the parent compd for several weeks in methanolic ammonium polysulfide (Ref 2c)

Ethanemercarbide Hexaiodide, $(IHg)_3C.C(HgI)_3$; mw 1989.20, OB to CO_2 –8%, red-yel cryst; may be prepd by treating one mole of the parent compd with 6 moles of EtI for 60 hrs at 90°C (Ref 2b)

Ethanemercarbide Hexachloride, $(ClHg)_3C.C(HgCl)_3$, mw 1440.41, OB to CO_2 –11%; wh cryst, decomp on heating, sl sol in water, dil HCl; may be prepd by heating the parent compd with HCl soln (Ref 2b)

Dichloroethanemercarbide, $C_2Cl_2Hg_6O_2$; mw 1330.59, OB to CO_2 –9.6%; may be prepd by heating the parent compd in a KCl soln, or treating the parent with 10% HCl at RT (Ref 2b)

Dichloroethanemercarbide Diammoniate, $C_2H_6Cl_2Hg_6N_2O_2$; mw 1364.65, OB to CO_2 –12.9%; may be prepd by heating the dichloro derivative for 6 hrs at 40°C in ammoniacal NH_4Cl (Ref 2b)

Ethanemercarbide Dichlorate or *Dibromate*, explodes violently on heating or rubbing (Ref 2c)

Ethanemercarbide Diperchlorate, $C_2Cl_2Hg_6O_{10}$; mw 1458.59, OB 0%, wh powd, expl on heating or rubbing (with a green light); may be prepd by treating parent compd with $HClO_4$ (Ref 2b)

Ethanemercarbide Dipicrate, $C_{14}H_8Hg_6N_6O_{22}$; mw 1751.79, OB to CO_2 −14.5%; yel solid; stronger deton on heating than K picrate (Ref 2a)

Ethanemercarbide Dichromate, $C_2Cr_2H_4Hg_6O_{14}$, mw 1559.59, OB to CO_2 −1.0%; mild expln on heating (Ref 2c)

Following Refs were used for Ethanemercarbide and Derivatives:
Refs: 1) Beil **2**, 562–3 2) K.A. Hofmann et al, Ber a) **31**, 1904, 2217 (1898); b) **33**, 1329, 1332 (1900); c) **38**, 3654 (1905)

Ethanenitrile. See Acetonitrile in Vol 1 of Encycl, p A43

Ethanephosphorous Acid, Esters, $EtP(OR)_2$; prepd by treating $EtPCl_2$ with CH_3OH (or C_2H_5OH) in ether in the presence of pyridine or Et_3N in a CO_2 atm. The Me and Et esters are odoriferous liquids which inflame spontaneously on exposure to air when spread on filter paper. The *methyl ester*, $EtP(OMe)_2$, has bp 73.5–74.5° at 225mm, d 0.9515 at 20°, n_D 1.4210 at 20° and the *ethyl ester*, $EtP(OEt)_2$, has bp 58–60° at 38mm, d 0.9207 at 20° & n_D 1.4212 at 20°.
Ref: B.A. Arbuzov & N.I. Rizpolozhenskii, DoklAkadNauk **83**, 581 (1951) & CA **47**, 3226 (1953)

Ethanetetracetic Acid and Derivatives
Ethane-1,1,2,2-tetracetic Acid or *β,β-Bis-(carboxymethyl)-adipic Acid* (Called Äthan-1.1.2.2-tetraessigsäure in Ger), $(HO_2CCH_2)_2CH.CH(CH_2CO_2H)_2$; mw 262.21, crysts, mp 212–14°. Blood et al (Ref 2) investigated six methods for its synthesis and of these, the only one successful was the oxidation of 4,4-bis(tetrahydropyranyl). Different methods for prepn of acid were used by Runge (Ref 3) and then by Grundmann & Paul (Ref 4)

Runge & Goldann (Ref 5) prepd by treating its *tetrahydrazide* (mp 230.5° with decompn) with $NaNO_2$ and HCl at 8–10°:
Ethane-1,1,2,2-tetracetic Acid Hydrazide, called 3,4-Bis[(azidoformyl)methyl]hexane; diol Azide in CA 5th Decennial Formula Index, p 555F,

$$N_3OC.CH_2.CH.CH_2.CON_3$$
$$\quad\quad\quad

$(C_2H_5O_2C)_2C(NO_2).C(NO_2)(CO_2C_2H_5)_2$; mw 408.32, wh cryst, mp 65–66° (Ref 2), 74–75° (Ref 8). It was prepd by electrolysis of an 8% aq soln of $C_2H_5O_2C(C{=}NO_2^-NH_4^+)CO_2C_2H_5$; resistant to boiling alc KOH (Ref 2)

1,2-Dinitrosoethane-tetracarboxylic Acid Tetraethyl Ester, $(C_2H_5O_2C)_2C(NO).C(NO)(CO_2C_2H_5)_2$; mw 376.32, oil; prepd by electrolysis of a soln of a mixt of $(C_2H_5O_2C)_2C{=}NOH$ and its C=NONa salt (Ref 3)

Ethane-tetracarboxylic Acid Tetrahydrazide, $(H_2N.NHOC)_2CH.CH(CONH.NH_2)_2$; mw 262.2, N 42.7%, wh cryst, mp 205–210° dec, sl sol in org solvs; first prepd by Tiemann by refluxing 50g of the tetraethyl ester with 42g of hydrazine hydrate in alc for two hrs (Ref 9)

Ethane-tetracarboxylic Acid Tetrazide, $(N_3OC)_2CH.CH(CON_3)_2$; mw 306.17, OB to CO_2 –47%, N 54.9%, wh cryst, expl when contacted with a flame. It was prepd from the tetrahydrazide tetrahydrochloride in dil chilled HCl by addn of aq $NaNO_2$, at best in 25% yield and only from no more than 3g of starting material (Ref 5)

Refs: 1) Beil **2**, 331–332, (699–700) 2) C. Ulpiani, O. Gasparini, Gazz **32** II, 236 (1902) 3) C. Ulpiani & G.A. Rodano, Gazz **36** II, 81 (1906) (Dinitroso) 4) H. Tiemann, "Über die Hydrazide der s-Äthan tetracarbonsäure", Inaug-Dissertation, Heidelberg (1911) 5) T. Curtius, JPraktChem **94**, 364, 367 (1916) (Ester, hydrazide, azide) 6) E. Philippi & J. Hanusch, Ber **53**, 1300 (1920) (Anhydride) 7) C. Mannich & E. Ganz, Ber **55**, 3509 (1922) 8) F. Allsop & J. Kenner, JChemSoc **1923**, 2315 9) S. Coffey, Rec **42**, 399 (1923) (Hydrazide) 10) H. Staudinger & W. Kreis, Helv **6**, 324 (1923) (Anhydride) 11) E. Philippi & R. Seka, Monatsh **45**, 273–9 (1924) (Ester) 12) A. Michael & J. Ross, JACS **55**, 3693 (1933)

Ethane-tetraurethane and Derivatives

Ethane-tetraurethane, called *Acetylene-tetraurethane* in Ref 4; (called Äthantetraurethan or 1.1.2.2-Tetrakis [carbäthoxy-amino]-äthan, in Ger), $(C_2H_5O_2CNH)_2CH.CH(NH.CO_2C_2H_5)_2$; mw 378.38, N 14.81%, wh ndls (from abs alc), mp 260° dec (Ref 6), 268° (Ref 2), 276–78° dec (Ref 7), 286–87° (Ref 5), subl 240° (0.2 torr), sol in alc and in ether (Ref 2), insol in alc, dioxane, hexane (Refs 3, 5, & 7). It was first prepd by Curtius (Refs 1 & 2) by refluxing an ether soln of 1,1,2,2-Ethane tetracarboxylic acid Tetrazide with abs alc; later prepd by refluxing at 100° a mixture of ethyl carbamate, 30% aqueous glyoxal and hydrochloric acid, in 49% yield (Refs 3, 5 & 7); most recently prepd by refluxing glyoxal hydrate with ethyl carbamate and boron trifluoride etherate in benz, in 68% yield (Ref 7). Ref 7 states that the compd is incorrectly identified in Ref 3 as $C_2H_5O_2CN{=}CH.CH{=}NCO_2C_2H_5$. CA **25**(1931) Subject and Formula Indices for Ref 3 list the compd as $C_2H_5O_2CNHC{\equiv}CNH.CO_2C_2H_5$ and Ref 5 agrees with Ref 3. "Sadtler Standard Spectra, Midget Edition", Sadtler Research Labs, Philadelphia, Pa (1962), No 3888 agrees with CA, and Ref 6 agrees with Ref 2 but gives no reasons. Neither of Refs (3, 5, 7) appears to be aware of Refs (1, 2).

Refs: 1) Beil **3**, (12) 2) T. Curtius, JPraktChem **94**, 369 (1916) 3) H. Pauly & H. Sauter, ChemBer **63**, 2068 (1930) 4) ADL Synthesis HE's, 2nd Rept (1951), pp 51 & 53 (Gives incorrect Vol and p of Ref 2) 5) N.G. Gaylord, JOC **20**, 546 (1955) 6) BASF FrP 1128263 (1957), ChemZtr **130**, 1605 (1959) 7) P.M. Quan, JOC **33**, 3937–38 (1968)

Ethane-tricarboxylic Acid and Derivatives

Ethane-tricarboxylic Acid or *Metriol* (Mtr), (Äthan-tricarbonsäure, in Ger), $HO_2C.CH_2.CH(CO_2H)_2$; mw 162.1, OB to CO_2 –74%. This subst is listed as a parent compd for the triazide, although it was not used to prep it. It was, however, used for prepn of its trinitrate

Ethane-tricarboxylic Acid Triazide, $N_3OC.CH_2.CH(CON_3)_2$; mw 237.14, N 53.15%; viscous oil, expl violently on heating; prepd by Curtius by treating trihydrazide diammonium of ethane-tetracarboxylic acid with HCl, followed by $NaNO_2$

Refs: 1) Beil **2**, 812 & (681) (parent); **2**, 321 (triazide) 2) T. Curtius, JPraktChem **94**, 370–71, 380 (1916)

Ethane-1,1,1-tris(hydroxymethyl)Trinitrate (Popularly called **Metriol Trinitrate (MtrT)**, also, variously Methyltrimethylolmethane Tri-

nitrate; Nitropentaglycerin, Pentaglycerol Trinitrate, Methyl-tris(hydroxymethyl) methane Trinitrate, $CH_3C(CH_2ONO_2)_3$; mw 255.16, NO_2 54%, N 16.47%, OB to CO_2 −34.5%; heavy, colorless, odorless oil with flat taste; fr p −60°, sp gr 1.4685 at 20/4°, n_D 1.4760 at 17.5°, n_D 1.4748 at 25°. Sp mass given as 1.451 (Ref 8). Solubility is 0.5g/l in w.

Accdg to Refs 1a & 8a, MtrT was developed in Italy before WWII by Bombrini-Parodi-Delfino and then adopted by the Germans

The following method of prepn was used during WWII at the Krümmel Fabrik of Dynamit AG:

a) 50kg of finely pulverized metriol was slowly fed with stirring by means of a worm screw into a stainless steel nitrator contg 175kg of mixed acid (65% HNO_3 − 35% H_2SO_4) maintained at 20° C. Formation of lump was avoided because this could lead to overheating and decompn of metriol and acid

b) After 20 mins of nitration, 15 mins were allowed for separation of the oil from acid

c) The separated oil was washed, first with warm water, then with soda ash soln and finally with water. The temperature during all the washings was maintained at 40° because at a low temp the mixture was too viscous. The soda ash wash lasted 20 mins. The yield was 200 parts of MtrT per 100ps of Mtr

d) The washed oil was taken to a storage tank from which it was withdrawn when needed for prepn of "Rohpulvermasse" (rawpaste), serving for prepn of proplnts (See Note, below)

Ger technical MtrT was a heavy oil, practically insol in w, with N=16.00 to 16.32%, sp gr 1.460 at 20°, stability by Abel Test at 82° − 20 mins, decompn temp ca 182°, impact sensitivity with 2kg hammer 4cm, calorific value 1270kcal/kg (water in liq phase) and volatility less than for NG. It was used in some smokeless proplnts and as a replacement for NG

Note: Rohpulvermasse, Raw Propellant Mass or *Rawpaste* was a mixture of water-wet NC with an expl oil, such as MtrT NG, DEGDN, TEGDN, etc. Such wet mixt was safely transported when a smokeless proplnt plant was not located adjacent to the plants manufg NC and organic nitric esters. Prepn of Rohpulvermasse and of propellants as practiced during WWII at Krümmel and Dunneberg Plants of Dynamit AG is described in Refs 1a, pp 6, 10 & 65 and in Ref 8a, p Ger 169

Other props of purified Metriol Trinitrate: impact sensitivity 0.59kg/m for 50% detons (Ref 2). Dipole moment 3.39D at 25° (Ref 10). Vapor pressure over 26–72° given by Clausius-Clapeyron eqn: $\log p = A - B/T$ where A = 14.6237, B = 4603.4; calcd heat of vapzn given as 21kcal/m (Ref 11). Stability towards alk hydrate given as: sp rate of hyd = 6.9×10^{-4}/M sec, at 30° in 90% alc (v/v) with 0.08–0.15M NaOH (Ref 13). Min film thickness for propagation of HV deton (7100m/s) given as 0.16 inch, for LV deton (1500m/s) given as 0.01 inch (Ref 14). It was prepd from the alcohol with nitric acid at 10° in 90% yield, with nitric-sulfuric acid at 10° in 93% yield, and with nitric-sulfuric acid at 70° in 98% yield (Ref 2); also from the alcohol with 98% nitric acid in glacial acetic acid at RT in 90% yield (Ref 4). Infrared spectrum (Refs 5 & 9) shows the three major peaks at 6.04, 7.87 and 11.9μ. Addnl drop weight test data given in Ref 3. Heats of expln and formation given in Ref 7. Gap sensitivity given in Ref 12; patented for use with 1,3-diol-2,2-dimethyl-pentane dinitrate as main expl ingredients in a commercial blasting agent (Ref 6)

Refs: 1) Beil – not listed 1a) O.W. Stickland et al, "General Summary of Explosive Plants", PB Rept **925** (1944), 15 & 61 2) R. Colson, MP **30**, 43–50 (1948) & CA **45**, 8249 (1951) 3) L. Médard, MP **31**, 131–43 (1949) & CA **46**, 11685 (1952) 4) R.E. Barnhart & R.M. Cavanaugh, USP 2545538 (1951) & CA **45**, 7139 (1951) 5) F. Pristera, AnalChem **25**, 844–56 (1953) 6) W.H. Rinkenbach, USP 2709130 (1955) & CA **49**, 14325 (1955) 7) P. Tavernier, MP **38**, 301–36 (1956) & CA **51**, 15952 (1957) 8) P. Tavernier, MP **39**, 253–96 (1957) & CA **53**, 1715 (1959) 8a) Fedoroff et al, PATR **2510** (1958), pp Ger 113-L & Ger 169 9) F. Pristera et al, AnalChem **32**, 495–508 (1960) 10) A.R. Lawrence & A.J. Matuszko, JPhysChem **65**, 1903–4 (1961) 11) A.L. Woodman & A. Adicoff, JChemEng Data **8**, 241–2 (1963) & CA **59**, 436 (1963) 12) F.C. Gibson et al, NASA Accession No N64-23112, Rept No AD 438019 Avail OTS, 11pp (1964)

& CA **62**, 8923e(1965) 12a) Urbański **2** (1965), p 197–98 (1,1,1-Trimethylolethane Trinitrate) 13) R.T.M. Fraser, Chem & Ind **1968**, (33), 1117–18 & CA **69**, 78867(1968) 14) J. Ribovich, AnnNYAcadSci **1968**, 152(1), 766–72 & CA **70**, 69726(1969) 15) W.L. Schwoyer, USP 3461007(1969) & CA **71**, 93221(1969)

ETHANOL AND DERIVATIVES

Ethanol, Ethylol, Ethyl Alcohol or *Alcohol* (Alcool in Fr, Alkohol in Ger and Alkogol' in Rus), CH_3CH_2OH; mw 46.07, colorless liq, sp gr 0.879 at 20°/4, fr p −114.5°, bp 78.4°, fl p of 95% alc 14°C(57°F), heat of combustion 327.6kcal/mole and heat of formation −66.4kcal/mole, miscible with w, eth, methanol & chlf; sol in many other org solvents. It is a good solvent for many expls and its mixture with eth dissolves NC of 12%N. Alcohol can be derived from ethylene either by direct catalytic hydration or by means of ethyl sulfate as an intermediate. It can also be obtd as a by-product of hydrocarbon synthesis from CO and H_2, and as a by-product of methanol synthesis from these gases. The oldest method of alcohol prepn is by fermentation of grains, molasses and carbohydrates. It can also be prepd by fermentation of sulfite pulp. Alcohol prepd from grains is known as *grain alcohol* and it is used in prepn of beverages (Refs 1, 11 & 12)

The strength of alcohol commonly used in the US is 95% (190 proof), while 96% is used in Europe. For prepn of perfumes the 70% alcohol is used. The so-called *absolute alcohol* (200 proof) is at least 99% and its use is restricted to analytical purposes or research. The so-called *industrial alcohol* is unfit to drink because it contains up to 5% methanol (wood alcohol) or benzene. Such alcohol is known as *denatured* and it is briefly described in Vol 3 of Encycl, p D63-R. Denatured alcohol is used as a solvent for expls, rocket fuels, rubbers, detergents, etc. Mixture of 1 part alcohol with 2 parts eth (described in Vol 5 of Encycl as Diethyl Ether) used as a solvent in dehydration of Pyrocellulose in manuf of single-base proplnt, such as described in Davis (Ref 2) and also on p C399 of Vol 3 of Encycl, under "Colloiding Agents and Colloidal Propellants"

US Spec JAN-A-463B, Interim AMD 1(Mu), 22 March 1967, gives the requirements and tests for ethyl alcohol for Ordnance use (See below)

Stettbacher (Refs 3 & 5), who obtd information from Amer journal "Engineering" of Aug 30, 1946 stated that Ger WWII 320-kg rocket was propelled by mixture of alcohol 32.4 parts by wt and liquid oxygen 67.6ps. Its heat of combstn was 2020kcal/kg and temperature of expln 2760°C. A similar mixture or possibly a mixture of equal parts of alcohol and liq oxygen, which weighed 8.1 metric tons, was used for propelling V2

In the paper published in Explosivstoffe, Stettbacher gives thermodynamic data for alcohol/oxygen and alcohol/hydrogen peroxide rocket fuels, the latter having only 1477kcal/kg as heat of combustion

Use of alcohol as an ingredient of liquid rocket fuels also was described by Krop (Ref 6) and by Warren (Ref 10)

Accdg to Grosse (Ref 9), a mixt of frozen alcohol with liquid oxygen can be made to detonate by a sharp blow with a hammer. When alcohol was frozen in liquid air and nitrogen allowed to boil off, leaving a mixt of alcohol and oxygen, the residual mixture was expl

Expln of mixtures of alcohol-nitrogen dioxide was discussed by Gray & Yoffe (Ref 4)

Protection against alcohol explns was discussed by Sauerteig (Ref 7)

Sax (Ref 13) indicates that air containing more than 1000 parts per million of ethyl alcohol is irritating to the eyes and upper respiratory system. He rates it dangerous as a fire or disaster hazard when exposed to heat or flame, and moderate as an expln hazard when exposed to flame

Refs: 1) Beil **1**, 292, (146) & [281] 2) Davis (1943), 299–305 3) Stettbacher (1948), 52–3 4) P. Gray & A. Yoffe, JCS **1950**, 3183–84 & CA **46**, 26(1952) 5) Stettbacher, Pólvoras (1952), 68 6) S. Krop, JetPropulsion **24**, 224 (1954) 7) H. Sauerteig, Branntweinwirtschaft **77**, 138–43(1955) & CA **49**, 9278–79(1955) 8) A. Stettbacher, Explosivstoffe **1956**, 25 & 29–30 8a) Sax (1957), 247 9) A.V. Grosse, ChemEngrgNews **35**, pp 12 & 14 (Aug 15, 1957) 10) Warren (1958), 21 & 25 11) CondChemDict (1961), pp 457-R & 458-L 12) Kirk & Othmer, 2nd ed, Vol 8 (1965), p 422 13) Sax, 3rd ed (1968), p 733

Use of Ethanol and Ether in the Manufacture of Single-Base Smokeless Propellants. Operations in manuf of single-base smokeless proplnts from Pyrocellulose, which is NC of N content 12.60±0.10% (described in Vol 2 of Encycl, p C103) are discussed in Vol 3 of Encycl, under "Colloiding Agents and Colloidal Propellants" on pp C398 & C399. Manuf of NC is described in Vol 2, p C102, while its stabilization for use in smokeless propellants is described in Vol 2, p C107

Specification requirements and tests for ethanol used in manuf of proplnts are in MIL-E-463B with Interim AMD 1 (MU), March 22 1963. This Spec is discussed as next item

Specification requirements and tests for ether, given in MIL-E-199A(1), are discussed in this Vol under Ether, Diethyl, whereas its prepn and props are given in Vol 2, pp D1233–D1235, under Diethyl Ether

A good description of manuf of single-base proplnts, including four illustrations, is given in Davis (1943), pp 299 to D306

Note: During the *dehydration* procedure described in Vol 3, p C399-L, it is required to determine ethanol and water in the cylindrical *block* removed from the press. In these detns a piece of block (cake) is dried in an oven at 100° to obtain "total volatile content", the water is detd by Karl Fischer Method and then alc is obtd by difference. Finally ether is added to make a colloiding mixture contg 2 parts of ether to 1 part alcohol by wt. The above tests are described in the items which follow

Ref: N.M. Liszt, private communication, Oct 1972

ETHANOL, Analytical Procedures

The section described below was compiled in collaboration with Mr N.M. Liszt, Analytical Chemistry Branch Chief, Propellants Lab, FRL, Picatinny Arsenal

Ethanol, US Military Specification MIL-E-463B (14 May 1962), entitled *Ethyl Alcohol (For Ordnance Use)* requirements and tests, supplemented by *Interim Amendment 1(MU)* 22 March 1967)

1.1 Scope. This Spec and Amd 1 cover the requirements for six grades of ethyl alcohol intended for Ordnance Use

1.2 Classification. Ethyl alcohol shall be of the following grades:

Grade 1 – 95% by volume without denaturant

Grade 2 – 94.9% by vol with denaturant

Grade 3 – 99% by vol (anhydrous) with benzene denaturant

Grade 4 – 95% by vol denatured with tertiary-butyl alcohol and benzyldiethyl(2,6-xylyl-carbamoyl) ammonium benzoate

Grade 5 – 95% by vol (See 3.1.5)

Grade 6 – 88.3% by vol (See 3.1.6)

2.1 Applicable Documents – Listed among them are:

a) Federal Spec **O-E-760** for Denatured Ethanol; b) **ASTM D1364**, "Water in Volatile Solvents (Fischer Reagent Titration Method)", copy of which can be obtd from the American Society for Testing and Materials, 1916 Race St, Philadelphia, Pa, 19103; and c) Code of Federal Regulations **49-CFR71-90**, "Interstate Commerce Commission Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles", available from the Superintendent of Documents, US Government Printing Office, Washington, DC, 20025

3. Requirements

3.1 Material. Ethanol, prior to the addn of dye or denaturant, shall be a clear, transparent, colorless liquid, having a characteristic odor, and shall not contain visible insoluble material, either before or after the addition of dye or denaturant. Table Et 1 lists compositions of alcohols intended for Ordnance uses

3.1.1 Applicable to Grade 1. It shall contain neither dye nor denaturant

3.1.2 Applicable to Grade 2. It shall be made by the addn of benzene to alc that complies with the requirements of Grade 1, or that has been recovered from the manufg operation in in which it will be used

3.1.3 Applicable to Grade 3. It shall be made by the addn of benzene to an anhydrous grade (99%) of alc contg neither dye nor denaturant

3.1.4 Applicable to Grade 4. It shall be made by the addn of 1/8 gallon of tert-butyl alcohol and 1/4 avoirdupois ounce of benzyldiethyl-(2,6-xylylcarbamoyl) ammonium benzoate to 100 gallons of ethyl alcohol that complies with the requirements of Grade 1. It shall be dyed with 1 part by wt of fuchsin dye to 500,000 parts by wt of alcohol

3.1.5 Applicable to Grade 5. It shall be made by mixing 100 gallons anhydrous (99%) alcohol with 5 gallons anhydrous methyl alcohol

3.1.6 Applicable to Grade 6. It shall be made by mixing 100 gallons of Grade 5 alcohol with 5 gallons of ethyl acetate and 1 gallon of methyl-isobutyl ketone

Table Et 1

Components	Grade 1	Grade 2	Grade 3	Grade 4	Grade 5	Grade 6
Ethyl alcohol, % by vol, min	95.0	94.9	99.0	94.9	95.0	88.3
Benzene, % by vol, max	0.01	0.75	0.75	–	–	–
Acidity, g/100ml as acetic acid, max	0.005	0.01	–	–	0.01	0.01
Aldehydes, mg/100ml, max	20.0	–	–	–	–	–
Non-volatile matter, % max	0.004	0.006	–	–	–	–
Organic impurities, % max	0.1	–	–	–	–	–
Methyl alcohol, % by vol, max	–	–	–	–	5.0	4.5
Ethyl acetate, % by vol, max	–	–	–	–	–	5.4
Methyl-isobutyl ketone, % by vol, max	–	–	–	–	–	1.5

3.2 Chemical and Physical Requirements. Alcohols shall comply with the requirements shown in Table Et 1, when detd as specified in the applicable sub-paragraph of 4.3

3.3 Taste. Grade 4 alcohol shall be distinctly bitter

4. Quality Assurance Provisions, which include:

4.1 General Quality Assurance Provisions

4.1.1 Contractor Quality Assurance System

4.1.2 Submission of Product

4.1.3 Government Verification

4.1.3.1 Surveillance

4.1.3.2 Product Inspection

4.2 Inspection Provisions, which include:

4.2.1 Lot Formation

4.2.2 Examination

4.2.3 Testing

4.2.3.1 Sampling

4.2.3.1.1 Containers

4.2.3.1.2 Tank Cars

are discussed on pp 3–5 of Spec MIL-E-463B

4.3 Test Methods and Procedures

4.3.1 Ethyl Alcohol Content. Determine specific gravity at 15.6/15.6°C (60/60°F) by means of a calibrated hydrometer, pycnometer or other standard method. Calculate the percentage of alcohol by volume from Table 2, attached to Spec MIL-E-463B

Note: Tables given in Lange and ChemRubberCo cannot be used because they are not as detailed in the range of 93.0 to 100% alcohol by volume (See also GasChromatographic Method of USI, described under 4.3.9 Ethyl Acetate)

4.3.2 Benzene Content. The test for benz in Grade 1 alcohol shall be made by the colorimetric method 4.3.2.1 and the tests for benz in Grades 2 and 3 alcohol shall be made by the spectrophotometric method 4.3.2.2 or 4.3.2.3

4.3.2.1 Colorimetric Method for Benzene Determination. In this method it is necessary to run simultaneous tests for three alcohols: Grade 1 alcohol (sample), Grade 1 95% alcohol contg 0.01% benzene (serving as a standard) and the blank consisting of 95% alcohol contg no benzene. All three of them must be treated in the same manner, as follows:

Mix in a 50ml beaker, (placed in an ice water bath) slowly, with mechanical stirring, 5 ml of white fuming nitric acid (contg no brown fumes) with 5ml concd sulfuric acid (sp gr 1.84), taking care not to allow the temp to go above 25°. Add from a small buret, dropwise, to the vigorously stirred and cooled mixed acid, 0.5ml each of the above mentioned alcohols from their own burets and pour the resulting mixture slowly into 40ml of cold water contd in the 2nd beaker, provided with a mechanical stirrer and placed in an ice water bath. The temp in the 2nd beaker shall not go above 25°. Transfer the mixture to a 125ml separatory funnel, add 20ml diethyl ether and shake cautiously for 1 min. Discard the lower layer

Add to the ether extract (contg Nitrobenzene, if any benzene is present) in the funnel 20ml distd water and shake for about 1 min to remove the bulk of residual acid. Discard the lower layer and add to the ether layer a 20ml soln, made by diluting 5ml of satd Na bicarbonate soln with 15ml of distd w. Shake and, after allowing the separation

of layers, remove the lower one carefully into clean beaker. Test the soln in the beaker with a universal indicator test paper for alkalinity. If the soln is acid, repeat the washing with fresh 20ml bicarbonate soln and test it as above. It must be alkaline, which is indicated by yellow to yel-brownish color. Discard both washings and transfer ether layer thru the mouth of the separatory funnel to a dry 50ml beaker. Rinse the funnel with about 10ml ether and run it into the same beaker. Place the beaker on a steam bath and evaporate the ether extract to near (but not complete) dryness. Cool the beaker and rinse its contents with 10ml benzene-free 95% ethanol into a dry test tube (16 by 150mm). Add 1 ml of butanone (methylethyl-ketone), shake and add 0.5ml dilute alcoholic NaOH soln, which was previously prepd by diluting 1 volume of aqueous NaOH soln (40g NaOH per 100ml distd w) with 10 vols of benzene-free 95% ethanol. Mix thoroughly and allow to stand for 5 minutes before testing it colorimetrically. The color of blank contg no benzene must be yellow to yellow-orange, while the color of "standard" contg 0.01% must be reddish. If the color of sample is not of greater reddish intensity than the standard, it is considered satisfactory

4.3.2.2 Spectrophotometric Method for Benzene Determination. This method is based on the fact that benzene has a characteristic absorption curve in the ultra-violet region. It is used for testing Grades 2 & 3 alcohols, each contg 0.75% benzene

4.3.2.2.1 Apparatus. Absorbance measurements shall be made using a spectrophotometer with ultra-violet accessories, matched 1 cm silica cells and a blue sensitive phototube in position. A Beckman Model DU Spectrophotometer has been found to be satisfactory

4.3.2.2.2 Standard Curve. Prepare standard solns of benzene in ethyl alc by pipetting 0.2, 0.5 and 0.8ml of reagent grade benzene respectively into labelled 100ml volumetric flasks. Dilute benz in each flask to the mark with benz-free, reagent grade alcohol (95% or absolute) and mix the solns. Pipet from each flask 10ml portions to three other 100ml vol flasks, respectively, dilute the solns in these flasks with reagent-grade methanol and mix thoroughly. Take the absorbance readings of these solns at a wave-length of 261 millimicrons using a methanol blank. Plot the absorbance readings against the concentration, and this should be a straight line passing thru the origin. Record all pertinent data and retain the graph for use with the particular instrument in future routine work after checking two points on the curve

4.3.2.2.3 Spectrophotometric Procedure. Pipet a 10ml portion of the sample to a 100ml volumetric flask, dilute to the mark with reagent-grade methanol and mix thoroughly. Fill one of the silica cells of the apparatus with the sample soln, whereas the 2nd cell is filled with pure methanol. Insert the cells in the cell holder and, after proper adjustment of instrument, take the reading at 261 millimicrons, while using a slit width chosen for best sensitivity. Read the quantity of benzene in the sample from the standard curve prepd in par 4.3.2.2.2

4.3.2.3 Alternate Method for Grade 2 and 3 Alcohols. Micropipet a 0.10ml portion of the sample to a dry test tube and add by means of a small pipet 5ml concd sulfuric acid contg 0.5% of formaldehyde soln which is not weaker than 37%. After shaking the tube, compare its color with that of a 0.10ml portion of alcohol contg 0.75% benzene and treated in the same manner as the sample. The color developed in the tube with sample shall not be darker than that developed in alc contg 0.75% benz. In case of question, doubt, or dispute, the spectrophotometric method described above shall be mandatory

4.3.3 Acidity. Mix 100ml of sample with 100ml of freshly-boiled distd w (free of CO_2), add 2 drops of bromothymol blue indicator (0.04% LaMotte or Taylor solns) and titrate with 0.01N Na hydroxide soln to a blue endpoint. Calculate the acidity as acetic acid as follows:

Acetic acid in g/100ml = 0.06VN,

where: V = ml of alkali used

N = normality of alkali

4.3.4 Aldehydes *(Preparation of Sample).* Transfer 120ml of alcohol sample to a 250ml Erlenmeyer flask, add about 20ml of distd w and a few grains of carborundum. Distill slowly into a 100ml volumetric flask to the mark, and mix

4.3.4.1 Aldehydes *(Procedure).* Transfer the above freshly distd alcohol from the 100ml volumetric flask to a 500ml flask, add about

100ml distd w and excess of 0.05N Na bisulfite, which is equivalent to approx 25ml of 0.05N iodine soln. After allowing to stand for about 30 mins with occasional shaking, add excess 0.05% N iodine soln and titrate this excess with 0.05N Na thiosulfate soln. Run a blank using the same quantities of 0.05N iodine and Na bisulfite as used in the sample. Calculate as follows:

Aldehydes, in mg/100ml = A x 1.1

where: A = difference in ml between the titrations of sample and blank of Na thiosulfate soln used

4.3.5 Nonvolatile Matter. Evaporate 100ml of the sample on a steam bath in a tared dish (preferably platinum) and dry the residue to constant weight in an oven, maintained at 105 to 110°C. The increase in weight of dish shall be calcd as percentage non-volatile residue. Calculate as follows:

Nonvolatile matter in g/100ml = B–A

where: A = tare wt of dish

B = wt of dish with residue

4.3.6 Organic Impurities. Prepare a standard soln by pipetting 0.1ml portion of reagent grade, isoamyl alcohol (isobutyl-carbinol) into 100ml volumetric flask and making up to the mark with reagent grade absolute alcohol. Both of the following, on this standard soln, and on the sample, shall be conducted using test tubes of uniform size. These tests are are especially intended for the detection of an excessive percentage of fusel oil in alcohols

4.3.6.1 Sulfuric Acid Method for Organic Impurities. Place 10ml of the sample in a large test tube and run sulfuric acid of sp gr 1.84 cautiously, with swirling, down the side of the test tube. The temp of the soln will rise to about 90°C. Place in an identical test tube 10ml of the standard soln and treat in the manner described above. Place both test tubes in an oven or bath at 65° for 18 hours and compare the colors developed in solns. The development of definitely darker color in the sample than in the standard soln shall be cause for rejection

4.3.6.2 Potassium Permanganate Method for Organic Impurities. Place in one of the two identical test tubes 10ml of the sample, while in the other tubes are placed 10ml of the standard soln of par 4.3.6. Adjust the temp in each tube to 25±0.2° and add to each tube one drop of soln contg 1g K permanganate per liter of distd w. Record the time and wait. Complete fading of the pink color in sample soln in less than 10 mins indicates the presence of more than 0.1% organic impurities. There shall be almost no pink color in the standard soln after standing for 10 mins

Note: Fusel oil can also be detd by the method 4.6.1.6 of Interim FedSpec O-E-00760C(TR-IR), which is as follows: Mix 10ml of alcohol to test with 5ml of distd w and 1 ml glycerin(USP) and allow to evaporate spontaneously from clean, odorless absorbent paper. The sample shall be considered as having passed the test if no foreign odor is perceptible when the last traces of alcohol evaporate

4.3.7 Water. Determine the percentage of water by wt in accordance with ASTM Method **D1364–64.** This method covers the detn of w in any proportion in volatile solvents (such as alcohol and ethyl acetate). It is not applicable in the presence of mercaptans, peroxides and appreciable quantities of aldehydes and amines. Although the ASTM method is similar to Karl Fischer method described in Vol 5 under DYNAMITE, to the method described in Bofors Manual and to the Method 101.5 of MIL-STD-286B, it differs from them in some details. For this reason the ASTM method is described here

To determine water, Fischer reagent (a soln of iodine, pyridine and sulfur dioxide in the molal ratio of 1:10:3) dissolved in anhydrous ethylene glycol monomethyl ether is added to a soln of the sample in anhydrous pryidine-ethylene glycol (1:4) until all w present has been consumed. This is evidenced by the persistence of the orange-red end point color; or alternatively by an indication of a galvanometer or similar current indicating device which records the depolarization of a pair of noble metal electrodes. The reagent is standardized by titration with water

a) *Instrument End Point* for this test is that point in the titration when two small Pt electrodes, upon which a potential of 20 to 50mv has been impressed, are depolarized by the addn of 0.05ml of Fischer reagent (6mg of water per ml) , causing a change of current flow of 10 to 20 μa which persists for at least 30 sec

Note: This end point is sometimes incorrectly called the "dead stop" which is the reverse of the above

b) *Color End Point.* During the titration, the

soln first turns yellow, then deepens and the end point is indicated by the sharp change to orange-red. This color must persist for at least 30 secs in order to indicate an end point

Apparatus

a) *Titration Vessel.* For color end point titrations, use a 100- or 250-ml volumetric flask fitted with interchangeable electrodes (Fig Et 1). Same flask may also be used for instrument end point and is particularly suitable for titrations at ice temps. For permanently mounted assemblies, the vessel should have a capacity of about 300ml, be provided with a tight-fitting closure, a stirrer, and a means of adding sample and reagents and removing spent reaction mixture. Such flasks are made by Rankin Glass Blowing Co, Martinez, Calif. It is desirable to have a means for cooling the titration vessel to ice temp (Fig Et 1)

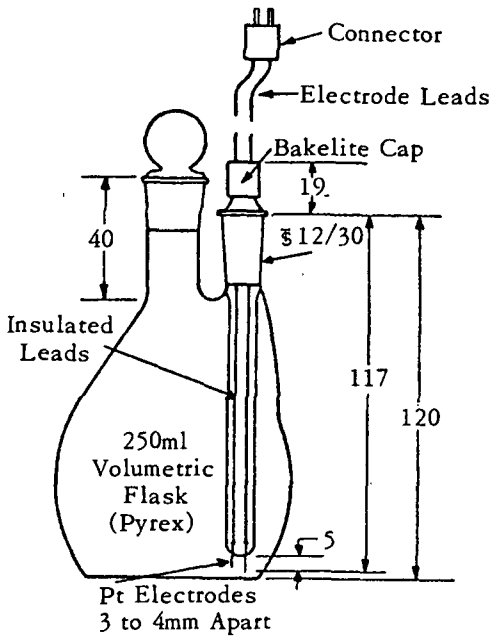


FIG Et 1 TITRATION FLASK ASSEMBLY

b) *Instrument Electrodes* are of platinum with surface equivalent to two No 26 wires 3/16 inch long. The wires should be 3 to 8mm apart and so inserted in the ve ssel that 25ml of liquid will cover them

c) *Instrument Depolarization Indicator* having an internal resistance of less than 5000 ohms and consisting of a means of impressing and showing a voltage of 20 to 50mv across the electrodes and capable of indicating a current flow of 10 to 20μa by means of a galvanometer or radio tuning eye circuit, similar to the "Aquatrator" of Precision Scientific Co, shown in Fig 5 and described in Vol 5 under DYNAMITE, "General Methods of Analysis Applicable to Commercial Blasting Explosives Including Dynamites" or "Fisher Titrimeter", described in catalogs of Fisher Scientific Co, Pittsburgh, Pa

d) *Buret Assembly* for Fischer reagent, consists of a 25- or 50-ml buret connected by means of glass (not rubber) to a balloon contg the reagent. Several types of automatic dispensing burets are on the market. Of these, the type similar to Catalog No J-821 of Scientific Glass Co, Bloomfield, NJ or Catalog No 750 of Eck & Krebs, NY, has been specifically designed for this analysis and presents the minimum contact of reagent with stopcock lubricant. One of such burets: "Macklett Automatic Burette" is shown in Fig 4 under DYNAMITE

Since the Fischer Reagent loses strength when exposed to moist air, all vents must be protected against atm moisture by means of drying tubes contg "Indicating Drierite". All stopcocks and joints should be lubricated with materials not particularly reactive with the reagent, such as Apiezon N (James G. Biddle & Co of Phila, Pa); High Vacuum Silicone Grease (Dow Corning Co, Midland, Mich); or Sisco 300 (Swedish Iron & Steel Co, New York, NY)

Reagents:

Unless otherwise indicated, it is intended that all reagents conform to the specs established by the Committee on Analytical Reagents of the ACS, where such specs are available; otherwise the best available grade must be used

a) *Fischer Reagent* (1ml equivalent to 6ml water). For each liter of soln, dissolve 133$\pm$1g of I_2 in 425$\pm$2ml anhydrous (less than 0.1% water) pyridine in a dried glass stoppered bottle. Add 425$\pm$2ml anhydrous (less than 0.1% water) ethyleneglycol monomethyl ether, cool to below 4°C in an ice bath and introduce in the soln gaseous sulfur dioxide, dried by bubbling thru concd sulfuric acid (sp gr 1.84). Determine the amt of sulfur dioxide added by measuring the loss in wt of the sulfur dioxide cylinder (102$\pm$1g).

Alternately, add 70ml of freshly drawn liq sulfur dioxide in small increments

b) *Sample Solvent.* Mix 1 vol of anhydrous (less than 0.1% water) pyridine with 4 vols of anhydrous (less than 0.1% water) glycol and keep it in a glass-stoppered bottle

Note: If adequately dried reagents cannot be procured, they can each be dried by distillation thru a multiple-plate column, discarding the first 5% of material distilling overhead and using 95% remaining

c) *Standardization of Fischer Reagent.* Standardize it daily by either the color or instrument end point method, proceeding as follows:

Add 25ml of the above "sample solvent" to titration flask of Fig Et 1 and titrate with Fischer reagent to orange-red end point. Add to the solvent, thus titrated, instead of the sample, 0.15 to 0.18g distd w from a weighing pipet. Complete the titration with Fischer reagent as described below under "Titration of Sample"

Calc the equivalency factor of the reagent by means of the following equation:

Equivalency Factor, F = A/B

where:

A = mg H_2O added to "sample solvent", and

B = ml of Fischer reagent required for titration

d) **Titration of Sample**

It is essential to avoid changes in water content of the material during sampling operations. Errors from this source are particularly significant in the analysis of materials having low water or having the tendency to readily absorb moisture, such as absolute alcohol. It is necessary to limit all possible contact of the sample with air in transferring the sample into the titration vessel

Procedure:

Introduce 10 to 25ml of the anhydrous "sample solvent" (pyridine-glycol, 1:4) into the titration vessel (Fig Et 1), making sure, if an instrument end point apparatus is used, that the electrodes are covered by the amt of "sample solvent" introduced. If the color end point is also to be used, introduce 10 to 25ml sample into the 2nd titration flask

Adjust the stirrer to provide adequate mixing without splashing and titrate the mixture in the 1st flask with Fischer Reagent to the instrument end point, while the 2nd flask is titrated from the 2nd buret to the orange-red color end point

To each of the flasks add an amount of sample accdg to its water content, as follows: 25ml if water is assumed to be below 0.5%, 10ml for 0.2 to 2.5% water content and 0.15 to 0.18g sample for 2.5 to 10% water content

Continue titration of the 1st flask to the instrument end point, while the 2nd flask is titrated to the color end point

Calculation:

$$\text{Water Content, \%} = \frac{V \times F}{10 \times W}$$

where:

V = ml of Fischer Reagent required for sample

F = equivalency factor for Fischer Reagent, in mg of water per ml of Reagent, and

W = weight of sample in grams

4.3.8 Methyl Alcohol. The test is listed but not described in MIL-E-463B, but the following description is given in Interim Federal Specification, O-E-00760C (TR-IR), August 7 1970 under Test 4.6.1.5 on pp 6–7

Preparation of Reagents:

Potassium Permanganate Solution – 100ml of an aqueous soln contg 15ml of 85% phosphoric acid and 3g of potassium permanganate. Renew reagent every 4 weeks

Chromotropic Acid Solution. A 5% aqueous soln of the acid (1,8-dihydroxynaphthalene-3,6-sulfonic acid) or its Na salt

Procedure for Determination of Methyl Alcohol. Dilute a portion of the alcohol sample with distd w to make the ethanol content 5 to 6%. Pipet 2ml of the permanganate soln into a 50ml volumetric flask, chill in an ice bath, add 1 ml of the diluted alc sample and allow to oxidize in the ice bath. Decolorize the oxidized sample with a small amt of dry Na bisulfite and add 1 ml of the chromotropic acid reagent. Rotate the flask with a swirling motion, while adding by pipet 15ml of concd sulfuric acid, and place the flask in hot water (60–70°) for 15 mins. Remove the flask, cool to RT and make up to 50ml with distd w. Compare the color of the soln with that of a standard (95% ethanol, contg 0.1% methanol), which was subjected to the above procedure. The sample passes the test if the depth of purple color is less than that of the standard. If the color is less than light straw, no methanol is present (See also Gas Chromatographic Procedure of USI described under 4.3.9 Ethyl Acetate)

4.3.9 Ethyl Acetate. The test is listed but not described in MIL-E-463B. It is stated that "ethyl acetate content shall be determined by using a suitable method that has been approved

by the contracting officer''. As such statement does not help to solve the problem, we asked Mr N.M. Liszt to try to get the description of the method. He was able to obtain it thru the courtesy of Mr Michael Toto, Office Manager of US Industrial Chemical Co, NY 10016, who communicated the method to Mr Anthony Taschler of Picatinny Arsenal. The method is gas-chromatographic and is used by Industrial Chemical Co for detn of Ethyl Acetate, Ethyl Alcohol, Methyl Alcohol and Methyl-iso-butyl Ketone (MIBK). General procedure for gas chromatography is described in Vol 3 of our Encycl, pp C293-R & C294-L. Following is outline of method used by USI

A quantitative gas chromatographic method for the denaturants in Filmex C can be made under the following conditions:

Instrument – Varian Model 2800 chromatograph
Detector – Flame ionization
Attenuation – As noted on chart
Sample size – 1 μl
Range – 10^{-10} amps/mv
Column – 6' x 1/8'' Chromosorb 102
Program – Start 140°C, hold for 4 min; heat at 10°C/min to 195° and hold
Run is complete in 23 minutes

Calculation – Compare peak areas (or peak heights) of the unknown to those of a standard of similar composition to determine quantitative results

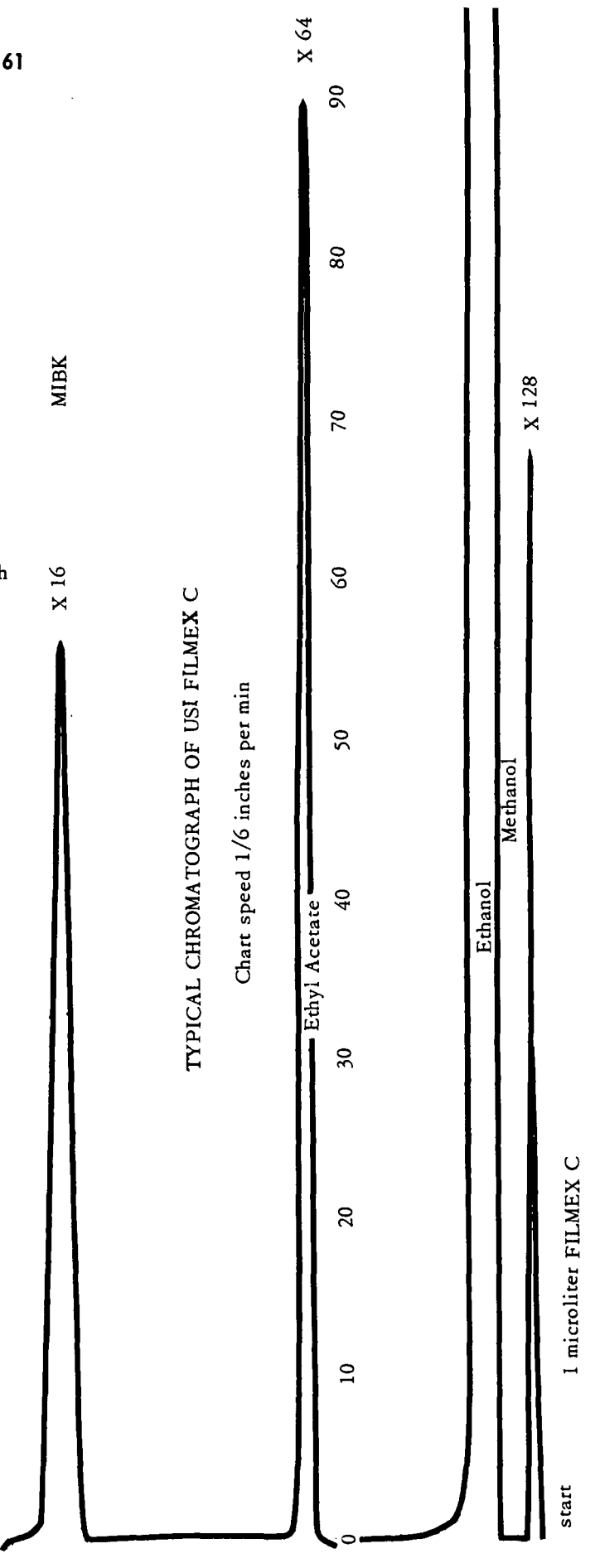
Figure shows a typical chromatogram of of Filmex C run under the above conditions

The chromosorb column will give good separation of the components under various temperature programs. The initial temperature can be below 100°C to give a wider peak for methanol if a high count integrator is not available. If desired, an internal standard can be used. Under the conditions described, benzene can be used

A suggested procedure would be equal volumes of the sample and a 4% (by volume) benzene in pure ethanol. This is easily set up using a G.C.-Computer system

Note: Ethyl Acetate can also be detd by Standard Method ASTM Designation D1617–69, described here under Ethyl Acetate

4.3.10 Methyl-isobutyl-ketone (MIBK). The test is listed but not described in MIL-E-463B, but it seems that the Test 4.6.1.8 for determination of Acetone and other ketones, described



in Interim Fed Spec O-E-00760C (TR-IR), 7 Aug 1970 can be applied here

Procedure: To 1 ml of the alcohol to test, add 4ml of distd w and 10ml of mercuric sulfate test soln (Denigès Reagent), prepd by mixing 5g of yellow mercuric oxide with 40ml distd w, followed by 20ml concd sulfuric acid, added slowly with stirring. Finally 40ml of distd w are added and the mixture is stirred until complete soln is attained. Then the soln is heated on a bath of boiling water and, if no ppt appears within 2 mins, the sample is considered as having passed the test. (See also Gas Chromatographic Procedure of USI, described under 4.3.9 Ethyl Acetate)

5.1 Packing. Ethanol shall be furnished in 5-gallon or 55-gallon metal containers as described on pp 10 and 11 of Spec MIL-E-463B

6.1 Intended Use:

Grade 1 – For use in the manuf of MF (Mercuric Fulminate), Smokeless Propellant (where a denaturant is not required), small arms primers, and optical elements

Grade 2 – For use in the manuf of Smokeless Propellant, ether, ethylene, and shellac mixtures

Grade 3 – For use in shellac mixtures for ammunition

Grade 4 – Denatured alcohol for general use where the grade in Federal Spec O-E-760 is not suitable for use

Grade 5 and Grade 6 are used in the mixing and manufacture of pyrotechnics (N.M. Liszt of Picatinny Arsenal; private communication)

Note: Benzyldiethyl(2,6-xylylcarbamoyl) ammonium benzoate may be purchased from Walker Chemical, Inc, 22 West First St, Mount Vemon, NY or other source

Ethanol Determination by Estimation of Its Hydroxyl Group, Using Various Esterification Procedures

Following methods are described in Std-MethodsChemAnalysis **2A**(1963), pp 455–460:

Esterification with Acetic Anhydride, Method 1: Uncatalyzed Reagent. It is based on the following reaction:

$$C_2H_5OH + \begin{matrix} CH_3CO \\ CH_3CO \end{matrix}\!\!>\!O \rightarrow CH_3COO.C_2H_5 + CH_3COOH$$

and is described on pp 455–59

Esterification with Acetic Anhydride, Method 2: Reagent Catalyzed with Perchloric Acid is described on pp 456–57. It is more rapid than Method 1

Esterification Using Phthalic Anhydride is described on p 458

Esterification Using Pyromellitic Dianhydride is described on pp 458–59. This method combines the advantages of esterification by acetic anhydride and by phthalic anhydride

In the same book, on p 462 is described "Colorimetric Method for Determining Trace Quantities of Hydroxy Compounds". This method developed by V.W. Reid & R.K. Trulove and described in Analyst **77**, 325 (1952) uses ceric ammonium reagent and is applicable to determination of small quantities of alcohol in aqueous or water miscible systems [Compare with J. Lamond, Analyst **74**, 560–61 (1949), listed here as Ref 5, under "Ethanol, Ether, Acetone and Water Determinations in Solvents Used in Manufacture of Smokeless Propellants, as Described in the Literature"]

Ethanol, Ether and Water Determination in Single-Base Propellants by US Army Munition Command Method T103.5 entitled: **Total Volatiles, Gas Chromatographic Method.** The method is described in MIL-STD-2688, 30 June 1971

1. *Scope*

1.1 This method is based on extraction of the solvents from the proplnt with a mixture of predried methyl ethyl ketone and secondary butyl alcohol (25/75% by volume). The method is directly applicable to proplnts contg NC of about 13.15% N. By controlling the ratio of extracting solvents, proplnts contg NC of lower N content (such as 12.60 or 12.00% N) can be analyzed by this procedure. For such proplnts, a lower MEK to sec-BuOH ratio should be used to prevent complete dissolvation

2. *Specimen*

2.1 The specimen shall consist of approx 10g of the proplnt with minimum atmospheric exposure in order to reduce loss of volatiles or adsorption of water. This procedure has been used successfully on the following single-base proplnts using original size: M-1 SP, M-1 MP, Ben ite, IMR & CBI and on crushed M-6. It should be applicable to other single-base formulations provided the specimen is cut to convenient size as specified in Method 509.3, entitled *Preparation of Propellant Samples*, described in MIL-STD-286B (1 Dec 1967). This

method is used for prepg sheet and granular proplnts for analysis. It is warned that procedures involving grinding should not be used for proplnts contg potentially hazardous oxidants, such as chlorates and perchlorates

Apparatus: a) Laboratory Mill (Wiley, Standard Model No 2G, or equivalent), equipped with a shield and an explosion-proof motor, Class 2

b) Beryllium alloy shears or razor blade;

c) Powder cutter (modified paper cutter, preferably with beryllium blade) and

d) Glass bottle, stoppered

Procedure for Sheet Propellant. Cut sheet into pieces appr 1/8-inch square, using razor blade or shears. Place cut pieces immediately in a glass bottle and stopper it

Note: If grinding is specified, make the proplnt brittle by chilling it in an ice bath prior to grinding as described for small grain proplnt

Procedure for Small Grain Propellant. Prepare the mill for grinding proplnt (of size 0.2g or less) by placing the 20-mesh screen betw the grinding chamber and the receiving container, and firmly clamp the rotor cover plate in position. After placing the shield in proper position, start the motor and grind few grains at a time, checking the temp of machine betw grindings. Place ground sample immediately in the bottle and stopper it. Allow sufficient time for the rotor and blades to cool before grinding addnl portions of proplnt

Procedure for Large Grain Propellant. Cut grains larger than 0.2g into slices approx 0.17mm thick, using the power cutter. Cut these slices into pieces approx 1/8-inch square and place them immediately in the bottle and stopper it

3. *Apparatus for Method T103.5*

3.1 Gas chromatograph (GC) equipped with a thermal conductivity detector and a one-millivolt recorder and/or integrator (or equivalent)

3.2 Column: eight-foot stainless steel, 1/4-inch outer diam tube contg 80–100 mesh Porapak Q

3.3 Erlenmeyer flasks, 125ml

3.4 Rubber stoppers, size 5½, solid

3.5 Syringe, 50 microliters

3.6 Flow meter, 10cc

3.7 Serum bottles, 30ml

3.8 Rubber stoppers for serum bottles

3.9 Pipets, 25 and 50ml, volumetric

3.10 Shaker, horizontal (for flasks)

4. *Materials for Method T103.5*

4.1 Acetone, reagent grade

4.2 Methyl ethyl ketone (MEK), certified reagent grade (Fisher Scientific Co, No M209, or equivalent)

4.3 Sec-butyl alcohol, reagent grade (Eastman Organic Chemicals Cat No 943 or equivalent)

4.4 Molecular sieves, type 4A, size 1/16 inch pellets

4.5 Ethyl alcohol, absolute, dried

4.6 Water, distilled

4.7 Diethyl ether, reagent grade, dried

4.8 Helium, commercial grade

4.9 Extraction soln, mixt of dry MEK 25 with dry sec-BuOH. It must be kept stored in contact with molecular sieves to keep dry

5. *Procedure for Method T103.5*

5.1 Preparation of Extraction Solvents

a) Dry solvents for extraction by addg approx one-inch layer of molecular sieves directly to the gallon containers of MEK and s-BuOH prior to mixing in proper ratio and allowing 2 days for the sieves to absorb the moisture

b) Mix MEK and s-BuOH in 25:75 ratio and add one-inch layer of molecular sieves to the container

5.2 *Preparation of Standard*

a) Pipet 25ml of the dry mixed solvent into a tared 30ml serum bottle

b) Stopper the bottle, weigh it with soln to the nearest 0.2mg and label

c) Using a syringe, inject thru the rubber stopper 0.10ml each of disted w, dry ethanol and ether, reweighing the bottle after each injection to the nearest 0.2mg. Be careful not to get any of the liquids on the stopper during injections

d) Record the wt of each component in the standard

5.3 *Testing*

a) Add approx 10g of proplnt, weighed to the nearest 0.2mg, to a 125ml Erlenmeyer flask and stopper immediately

b) Label the flask and record the wt of proplnt in it

c) Pipet 50ml of 25/75-MEK/BuOH solvent into the Erlenmeyer and immediately stopper it. Solvent must sufficiently cover the sample and if it does not, as with CBI proplnt, an addnl amt of solvent must be added

d) Place the tightly stoppered flask on the horizontal shaker at low speed and ambient condition for gentle shaking to swell the proplnt and extract volatile solvents from it

e) Extraction times are 2 hours minimum for M-1 SP, M-1 MP, Benite, CBI & IMR and 6 hours minimum for crushed M-6

f) Remove the flask from the shaker and allow 15 mins for settling the larger part of the solids

g) Check the instrument settings and make the necessary adjustments: injection point 160°C, oven 150°, detector 180°, bridge current 200 milliamperes, helium (carrier) flow 60cc/min and helium inlet pressure 50 psig

h) Without disturbing the settled solids in the Erlenm flask, remove 20μl of liquid (free of bubbles) by means of a 50μl syringe that has been previously rinsed with acetone and dried by forced air

i) Wipe the tip of syringe with a tissue and then draw up 1 μl of air

j) Immediately inj ect the sample into the gas chromatograph (GC) and allow the component peaks to evolve at their respective attenuations (See Fig Et 2)

k) The sequence of separation of the components will be: air, water, ethanol, impurity from MEK (only seen when GC is set at high sensitivity), ether, and extraction mixture (See Fig Et 2)

l) Depending upon sample concn, it may be necessary to change the attenuation in order to keep component peaks on the chart, but it is not necessary to keep MEK/s-BuOH peak on the chart

m) The attenuation should never be changed during the evolving of a peak and it is desirable to preattenuate so that a peak betw 30% and 95% of the chart can be obtd

n) At times it may be necessary to rezero the recorder and integrator after switching attenuator settings or after evolution of a component. True zero is when the integrator runs a straight line

o) Record on the chart the sample identification, the attenuation and the integrator reading of each component peak (See Fig Et 2)

p) Repeat paragraphs 5.3 (h) thru 5.3 (o) using the standard 5.2

q) Repeat paragraphs 5.3 (h) thru 5.3 (o) at an attenuation 1X using the MEK/s-BuOH soln to obtn the water correction (if necessary)

r) Calculation:

$$\% \text{ Alcohol} = \frac{(A)(B)(W_1)(100)(E)}{(C)(D)(W_2)}$$

$$\% \text{ Ether} = \frac{(A)(B)(W_1)(100)(E)}{(C)(D)(W_2)}$$

$$\% \text{ Water} = \frac{(AB-FG)(W_1)(100)(E)}{(CD-FG)(W_2)}$$

% TV (Total Volatiles) = % Alcohol + % Ether + % Water

where:

A = Attenuator setting for unkown sample (alcohol, ether or water)

B = Area of peak for unknown sample (alcohol, ether or water)

C = Attenuator setting for standard (alcohol, ether or water)

D = Area of peak for standard (alcohol, ether or water)

E = Ratio of solvent between sample and its standard (equals 2 when 50ml is used for sample and 25ml is used for standard)

F = Attenuator setting for water in the extraction solvent

G = Area of peak for water in the extraction solvent

W_1 = Grams of component per 25ml of standard

W_2 = Sample weight (10g)

Ethanol + Ether, as "Residual Solvent" Determination in Single-Base Propellants (US Ordnance Methods). Determine the percentage of total volatiles (Solvents + Moisture), then of moisture and subtract percentage of moisture from percent of total volatiles to obtain residual solvent

For determination of *Total Volatiles (Volatiles and Moisture)*, two methods can be recommended:

1. Vacuum Oven Method 101.1.2, Described in MIL-STD-286B (1 Dec 1967), is applicable to single-base, small-grain, proplnts

Procedure:

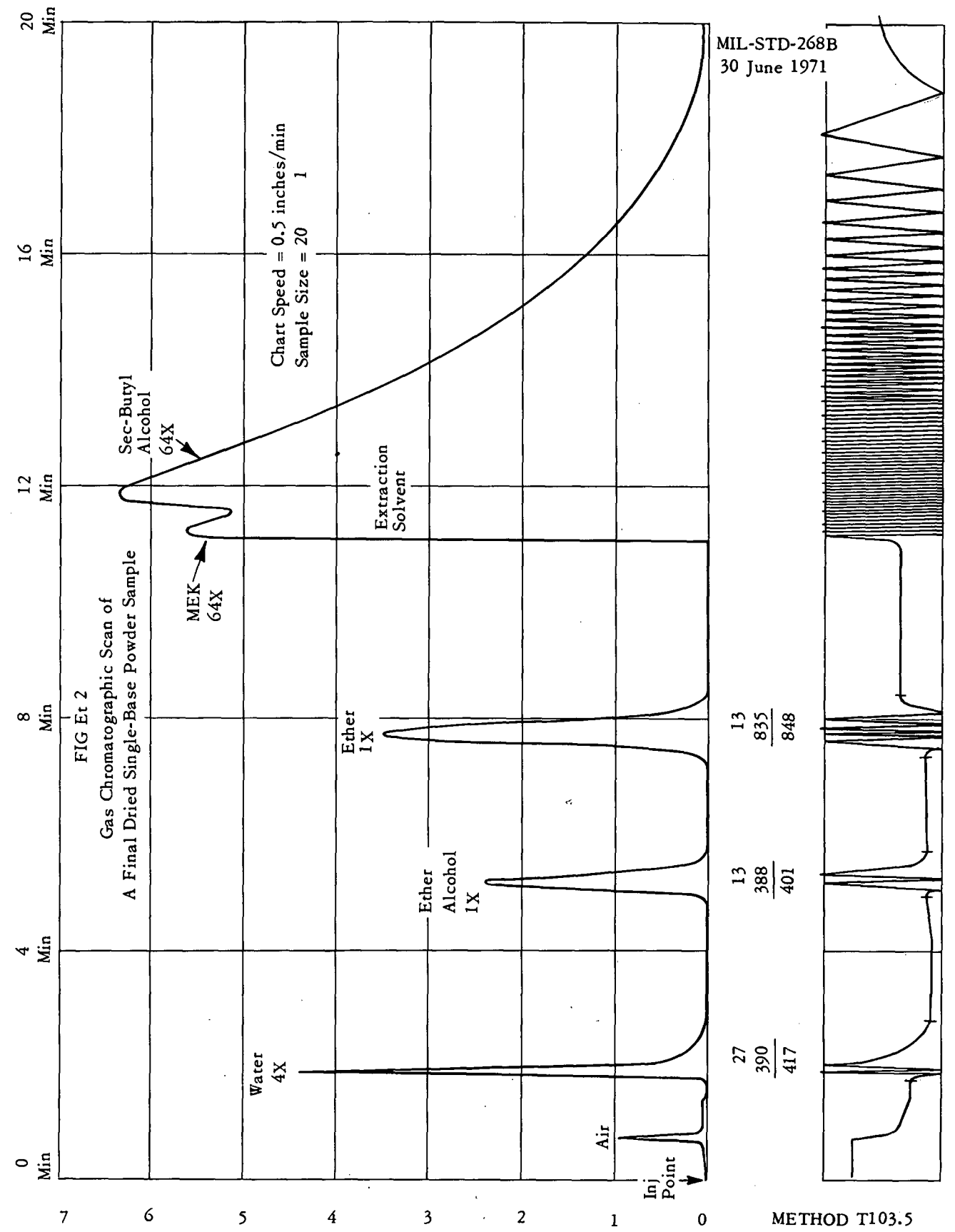
a) Tare to within 0.2mg a weighing dish, 30mm diam and 30mm deep together with a tight-fitting cover and place in it approx 10g sample of proplnt

b) After reweighing the dish with the sample, uncover it and place for 6 hours in a vacuum oven maintained at temp 55°±2°C and a pressure (absolute) of 80±10mm of mercury

c) Cool the dish with sample to RT in a desiccator (contg an indicating desiccant), replace the cover and reweigh the ensemble

Calculation:

$$\% \text{ Total Volatiles} = \frac{(W_1-W_2)\ 100}{W_1}$$


MIL-STD-268B
30 June 1971
FIG Et 2
Gas Chromatographic Scan of
A Final Dried Single-Base Powder Sample
Chart Speed = 0.5 inches/min
Sample Size = 20 1
Sec-Butyl Alcohol 64X
MEK 64X
Extraction Solvent
Ether 1X
Ether Alcohol 1X
Water 4X
Air
Inj Point
0 Min
4 Min
8 Min
12 Min
16 Min
20 Min
7
6
5
4
3
2
1
0
27 390 417
13 388 401
13 835 848
METHOD T103.5

where:

W_1 = Weight of sample before drying

W_2 = Weight of sample after drying

Note: This method is also briefly described in StdMethods of ChemAnalysis, Vol 2, Part B (1963), p 1294

II. Solution Evacuation Method 103.3.3, Described in MIL-STD-286B (1 Dec 1967 and 15 July 1969)

1. *Scope*

1.1 This method is used for determining the total volatiles content of either single-base, double-base, or triple-base proplnts

2. *Specimen*

2.1 The specimen shall consist of approximately 2g of the proplnt prepd with minimum atmospheric exposure in order to reduce loss of volatiles

3. *Apparatus*

3.1 Solution tubes (Fig Et 3)

3.2 Steel balls, 5/16-inch diam

3.3 Tubing, rubber, 5/16-inch bore, 3/16-inch wall

3.4 Vacuum line assembly (Fig Et 4)

3.5 Wire screen tray (Fig Et 5)

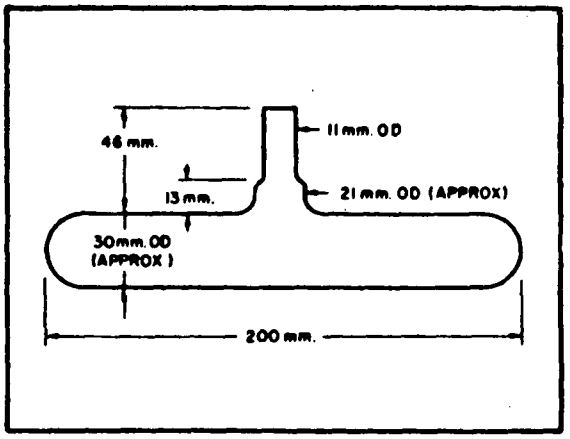


FIG Et 3 SOLUTION TUBE

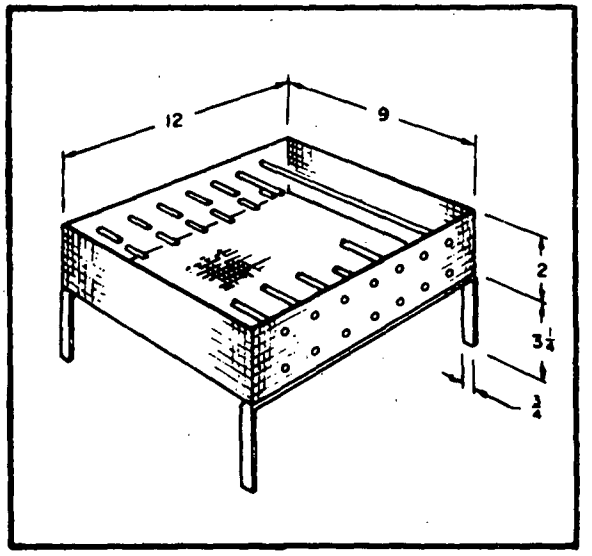


FIG Et 5 WIRE SCREEN TRAY

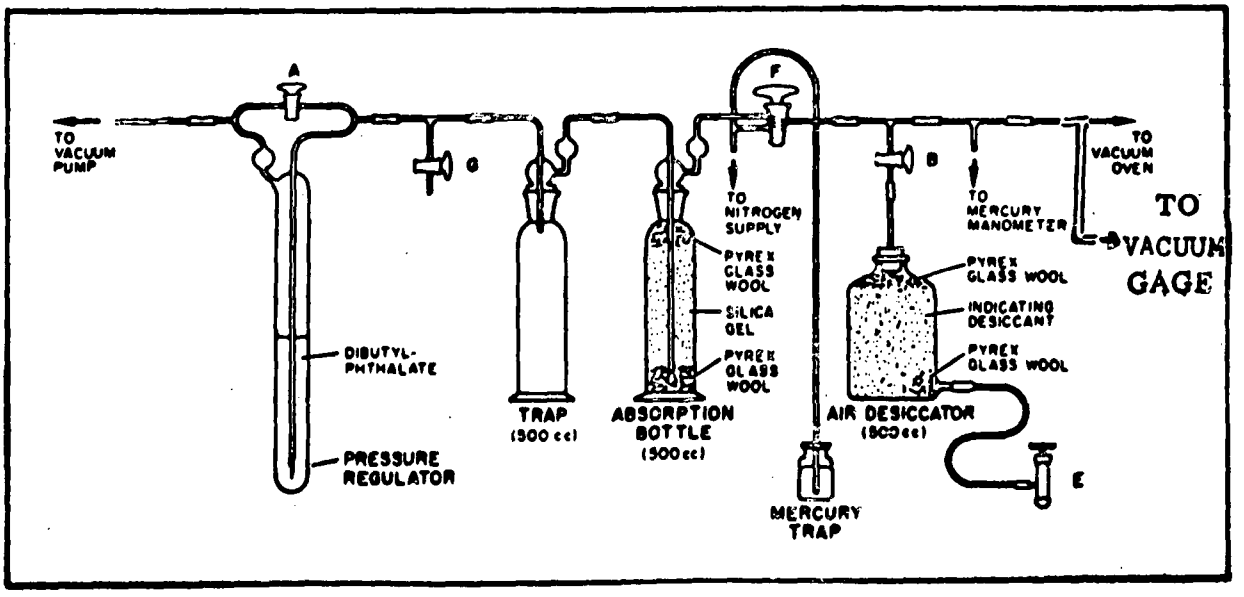


FIG Et 4 VACUUM LINE ASSEMBLY

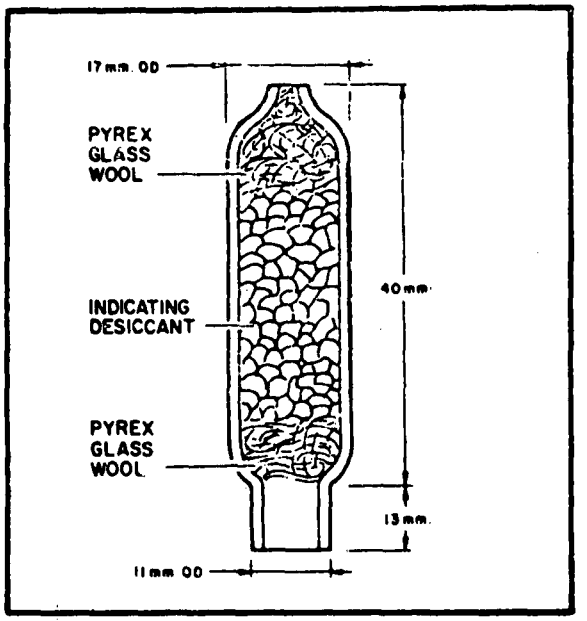


FIG Et 6 PROTECTIVE DRYING TUBE

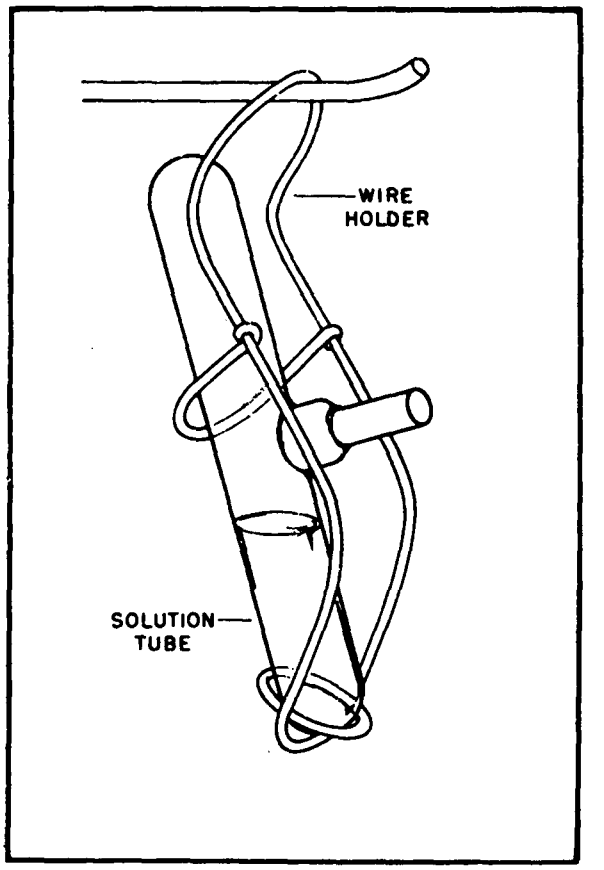


FIG Et 7 SOLUTION TUBE HOLDER

3.6 Protective drying tubes (Fig Et 6) containing indicating desiccant
3.7 Vacuum pump capable of maintaining a pressure (absolute) of at least 5mm of mercury
3.8 Wire holders for solution tubes (Fig Et 7)
3.9 Counterpoise (Fig Et 8)
3.10 Pyrex glass wool, or equivalent
3.11 Oven containing a rocking device with evacuation manifolds (Central Scientific Co, Cat No 95105-A modified by addition of a safety latch, or equivalent)

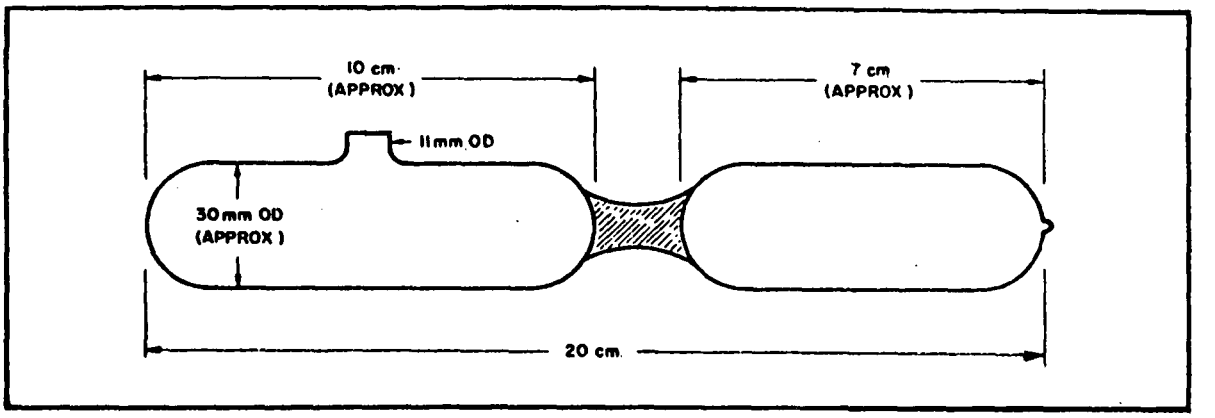


FIG Et 8 COUNTERPOISE

4. *Material*

4.1 Dibutylphthalate, prepared as follows:
a) Dissolve 1 g of diphenylamine in 100ml of hot dibutylphthalate
b) Pour this solution into 500ml of dibutylphthalate; stir vigorously
c) Add dibutylphthalate to make 1 liter; stir vigorously
d) Heat the solution for 2 hours at 145° to 150°C while bubbling dry air thru it
Note: The heating ensures that the solution will lose less than 10mg in 50ml during the solution-evacuation procedure, and may be omitted if previous tests show that is is unnecessary

5. *Procedure*

5.1 Prepare at least four solution tubes (two for the specimens, two for blanks) as follows:
a) Place 10 clean, dry, steel balls and 50ml of the dibutylphthalate reagent into each solution tube
b) Clean the inlet of each solution tube with absorbent cotton held by steel forceps, and insert a wad (approx 0.2g) of pyrex glass wool, using the forceps
Caution: Position the glass wool at such a point in the tube that it will not fall into the tube and will not touch the cork stopper that is to be inserted subsequently

5.2 Precondition the solution tubes as follows:
a) Using 5cm pieces of the rubber tubing, connect the solution tubes to the manifold of the rocking device in the oven, placing the tubes containing the blanks at opposite ends of the manifold. Plug or otherwise close unused manifold connections
Note: New connecting tubing should be precleaned by boiling in a 5% solution of sodium hydroxide for 10 to 15 mins, followed by a thorough rinsing, and drying at 100°C
b) Connect the vacuum line assembly (Fig Et 4) to the manifold of the rocking device and to the vacuum pump
c) On the vacuum line assembly (Fig Et 4) open stopcock A; close stopcocks B & G and needle valve E; and set stopcock F to connect the manifold to the vacuum pump
d) Adjust the oven temperature to 85°±1°C, start the rocking motor, and turn on the vacuum pump. Evacuate the tubes at a pressure of 5mm or less for 1½ hours
e) At the end of 1½ hours, stop the vacuum pump and rocking motor, and slowly open stopcock B. Then slowly admit dry air to the solution tubes by gradually opening needle valve E
Caution: If the air is admitted too rapidly, wads of pyrex wool may be drawn into the solution tubes
f) Wearing gloves, remove the solution tubes from the oven, leaving the connecting tubing attached to the tubes, and place them in the wire-screen tray (Fig Et 5). Immediately attach a protective drying tube (Fig Et 6) to the inlet of each solution tube to prevent the admission of atmospheric moisture Allow the tubes to cool to room temperature (approx 45 mins)
g) When the solution tubes have cooled, remove the protective drying tubes and connecting tubing, and insert No 1 cork stoppers
Caution: The solution tubes must be kept tightly corked hereafter to prevent absorption of atmospheric moisture by the very dry dibutylphthalate, except during evacuation in the oven and while being weighed

5.3 Clean and remove electrostatic charges from the solution tubes by wiping them with a wet towel and drying them with a clean lint-free cloth (without rubbing). Place the tubes in the wire-screen tray, cover them with a cloth to protect them from dust, and allow them to stand near the balance for at least 30 mins to attain equilibrium with the moisture content of the air
Caution: After the tubes have been conditioned they should not be touched with the fingers until they have been weighed. When handling the tubes, grip the inlet tube only, after covering the inlet with a tissue paper

5.4 Cut sheet and large grain proplnt as described in Method 509.3, paragraphs 4.1 and 4.3, respectively. Use small grain proplnt as received. Take at least two specimens

5.5 Remove the cork stoppers from two of the prepared solution tubes, and take out the wads of glass wool, using forceps. Add one of the accurately weighed specimens of the proplnt to each, using a small metal funnel (9mm O.D. outlet) to prevent adherence of the proplnt to the walls of the tube. Reinsert the wads of glass wool
Note: Proplnts soften and tend to gelatinize when added to dibutylphthalate solution. Therefore, the solution tubes should be rocked as soon as possible after the addition so that

the proplnt does not adhere to the walls of the tube and prevent the steel balls from moving

5.6 Support the tubes in wire holders (Fig Et 7) and weigh each solution tube (incl blanks) to within 1 mg, using the counterpoise (Fig Et 8) on the right-hand side of the balance

Note: The counterpoise approximates the weight, volume, and exterior surface area of a solution tube containing 10 steel balls and 50ml of dibutylphthalate. It should be kept standing near the balance, covered with a cloth to protect it from dust. *Do not wipe the counterpoise;* wiping will disturb its equilibrium with the prevailing temperature, pressure, and humidity

5.7 Shake the tubes, if necessary to loosen any grains of proplnt adhering to the walls of the tubes, and connect the tubes to the manifold of the rocking device in the oven. Plug all unused manifold connections

5.8 As soon as all the solution tubes have been placed in the oven (maintained at 85°±1°C), start the rocking motor

5.9 On the vacuum line assembly (Fig Et 4) check that stopcock F is turned so that nitrogen does not enter the vacuum line. Then open the valve on the nitrogen cylinder to allow a slow stream of nitrogen to escape thru the mercury trap

5.10 Close stopcocks B & G, and needle valve E, and open stopcock A. Then turn on the vacuum pump, and evacuate the soln tubes to a pressure of 1 mm of Hg or less

5.11 When the pressure has stabilized at 1 mm of Hg, turn stopcock F to admit nitrogen slowly to the soln tubes

Caution: Admit nitrogen slowly so that the glass wool or other foreign matter from the manifold or tubing is not carried into the soln tubes

5.12 Evacuate the tubes again, and refill with nitrogen by operating stopcock F

5.13 Turn stopcock F so that the nitrogen inlet is connected to the oven manifolds, open stopcock G to admit air to the vacuum line, and turn off the vacuum pump. Close stopcock G

5.14 Allow the soln tubes to rock until sample has completely broken up or a max of 15 hours at a temp of 85°±1°C

5.15 At the end of 15 hours, check that stopcock G is closed, and turn stopcock F to connect the oven manifolds to the vacuum pump. Open stopcock B and needle valve E, and start the vacuum pump

5.16 While observing the soln tubes thru the glass door of the oven, gradually close needle valve E to lower the pressure in the vacuum line assembly, being careful not to close the valve so fast that the soln in the tubes boils violently

Caution: Take approx 10 mins to lower the pressure to 5mm of Hg. If the soln boils violently, there is danger of mechanical loss of the soln, and a resulting error in the determination

5.17 When the pressure reaches 5mm, or less, close stopcocks A & B, and continue the evacuation for 2 hours at 85°±1°C, and at a pressure of 5mm of Hg or less

5.18 At the end of 2 hours, stop the rocking motor, open stopcocks A & B, very slowly open needle valve E to admit dry air, and stop the vacuum pump

Caution: If stopcock A is not open when the vacuum pump is shut off, the dibutylphthalate soln will flow out of the pressure regulator

5.19 Remove the soln tubes from the oven, and cool them, as described in paragraphs 5.2(f) and 5.2(g)

5.20 Condition the tubes as described in paragraph 5.3

5.21 Weigh the tubes, as described in paragraph 5.6 to determine the *loss* in weight of the tube containing the specimens, and the *change* in weight of the tubes containing the blanks

5.22 Calculate the percentage of total volatiles in each of the 2-gm specimens using the equation given below. The results of calculations must agree to within 0.10%. If not, repeat the analysis

$$\text{Percent total volatiles} = \frac{100\,(A + B)}{W}$$

where:

A = Decrease in weight of specimen tube

B = Change (average) in weight of blank tubes taken algebraically

W = Weight of specimen in grams

Note: The change in wt of the blank tubes usually is a decrease and the value substituted for B becomes a negative quantity. However, in some instances it is an increase (possibly as a result of humidity changes) and then the value substituted for B becomes a positive quantity. The blank values must agree to wi thin 2mg. If not, the analysis must be repeated

For determination of **Moisture** in single-base propellants, three methods can be used:

1. Electrolytic Hygrometer Method 101.4, Described in MIL-STD-286B (1 Dec 1967)

1. *Scope*

1.1 This method is used for determining the moisture content of small grain or flake proplnts. Single, double or triple base proplnts can be used. The principle of the method is measurement of the current required for electrolysis of water that has been volatilized from the specimen. Volatile compounds such as alcohols, amines and ammonia may also be electrolyzed. Compds that polymerize may eventually inactivate the cell

2. *Specimen*

2.1 The specimen shall consist of a 0.2–0.6g sample of proplnt weighed to the nearest 0.2mg

3. *Apparatus*

3.1 Solids moisture analyzer (Consolidated Electrodynamics Corp, 360 Sierra Madre Villa, Pasadena, Cal, or equivalent) (See Fig Et 9)

4. *Materials*

4.1 Nitrogen, extra-dry grade, in a cylinder with a pressure regulator to supply gas at 5psig. For calibration of the instrument, use accurately weighed samples of either 0.05 to 0.1 g of sodium tartrate dihydrate or 0.3 to 0.5g of potassium tartrate hemihydrate. On drying for 45 mins at 150°C, the sodium salt should give a moisture content of 15.66±0.05% while the potassium salt should give a moisture content of 3.83±0.02 percent

5. *Procedure*

5.1 Prepare the instrument for operation according to the manufacturer's directions

5.2 Place the weighed specimen in the sample boat, insert into the oven with the aid of tweezers provided with the instrument and close the oven

Caution: These steps must be done as rapidly as possible in order to minimize changes in moisture content

5.3 Turn the temperature and timer controls to the settings required in the applicable spec. When the test is completed, read the wt of the moisture shown on the dial and convert to grams

$$\text{Percent of water} = \frac{100A}{W}$$

where:

A = Weight of water shown on dial, g

W = Weight of specimen in grams

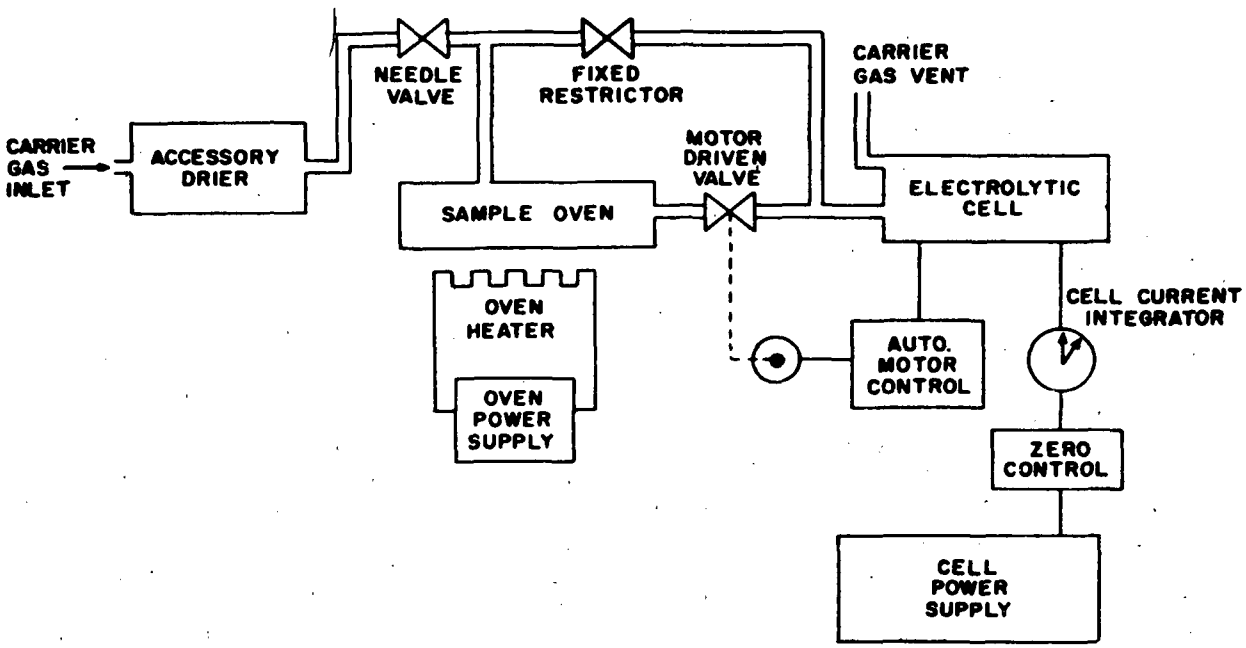


FIG Et 9 BLOCK DIAGRAM OF ELECTROLYTIC HYGROMETER

11. Moisture Determination in Propellants and Explosives by Distillation Method 102.1.3, Prescribed by MIL-STD-286B (1 Dec 1967). Although this procedure is similar to the one described under Dynamite in Vol 5, p D1620-R, we are describing it because it uses trichloroethylene (in lieu of carbon tetrachloride) as a solvent and a 100g (in lieu of 50g) sample. The same type of moisture tube (of Hercules Powder Co) is used in both cases (See Fig 1, p D1621-L in Vol 5)

1. *Scope*

1.1 This method is used for the determination of moisture content of small grain or finely divided cannon propellant. It is based on distillation of the moisture with a solvent that is immiscible with and heavier than water. Trichloroethylene is the preferred solvent, but carbon tetrachloride is also satisfactory. This method is not suitable for proplnts containing less than 0.2% moisture. The presence of hydrated compds may cause high results. The presence of finely divided aluminum may cause a reaction with the chlorinated solvent and, in such cases, tests should be made to demonstrate compatability of proplnt and solvent

2. *Specimen*

2.1 The specimen shall consist of approx 100g of the proplnt preferably as received. Weigh the specimen to within 10mg

Note: If the size of the proplnt as received would unnecessarily prolong the time required for the determination, the specimen may be cut as specified in Method 509.3. The cutting, weighing, and transferring to the stoppered balloon flask must be done rapidly to minimize the change in moisture content thru exposure to the atmosphere

3. *Apparatus*

3.1 Balloon flask, 500ml

3.2 Condenser (Allihn type, or equivalent), 400mm long (min)

3.3 Drying tube containing indicating calcium sulfate desiccant

3.4 Moisture tube (Kontes Glass Co, Cat No K-75500, or equivalent)

Caution: If ground glass joints are used, be sure that no proplnt is in the joints before making the connection

3.5 Hot plate (preferably steam or hot water heated)

4. *Materials*

4.1 Solvent: Trichloroethylene (preferably) or carbon tetrachloride

5. *Procedure*

5.1 Place the specimen in the 500ml balloon flask, and add 200ml of the solvent

5.2 Fill the graduated portion of the moisture tube with the solvent, and attach the tube to the flask. Attach the moisture tube to a dry condenser, and connect the drying tube containing the desiccant to the top of the condenser to keep out atmospheric moisture

5.3 Heat the flask so that the distillate falls from the condenser at the rate of two to three drops per second. Continue the distillation for the time indicated in Table Et 2

5.4 Remove the source of heat, and wash the water from the condenser into the moisture tube with 10ml portions of the solvent until the volume of the water layer in the measuring tube becomes constant, and no water droplets are observed in the condenser

Note: A total of 30 to 50ml of the solvent is usually required

5.5 Read the lower part of the top meniscus. Read the upper part of the lower meniscus

Note: To facilitate the reading of the menisci the surface of moisture tube and the condenser may be made water repellant by treatment with a suitable silicone preparation, such as Desicote (Beckman Instruments Inc). If water repellant material is used, read the point of contact of the top meniscus with the wall of the tube and the center of the lower meniscus

5.6 Determine the difference in the readings, and record the difference as the volume of water in the specimen

5.7 For each lot of solvent run a blank using exactly 2ml of water and apply any necessary corrections to the sample

5.8 Considering one ml of water as equal to one gram, calculate (by weight) the % of moisture in the specimen

$$\text{Percent water} = \frac{A - B}{W} 100$$

where:

A = Upper meniscus, ml

B = Lower meniscus, ml

W = Weight of specimen in grams

Table Et 2
Distillation Times

Propellant web size, inch	Grain condition	Distillation time, hours
Less than 0.025	Whole	3
0.025 to 0.040	Whole	5
0.041 to 0.060	Whole	10
0.061 to 0.090	Whole	16
	Sliced	6
Over 0.090	Whole	24
	Sliced	8

III. Moisture Determination in Propellants and Explosives by Karl Fischer Method 101.5, Prescribed by MIL-STD-286B (1 Dec 1967). Detailed description of Fischer method as applied to various substances is given in the following works:

1) A.R. Lusardi, "A Manual of Laboratory Procedures for the Analysis and Testing of Explosives and Pyrotechnics", Picatinny Arsenal, Dover, NJ (1962)

2) R.H. Pierson in StdMethodsChemAnalysis **2B**(1963), pp 1290–94

3) Vol 5 of this Encycl, under DYNAMITE

4) Anon, "Analytical Methods for Powders and Explosives", AB Bofors Nobelkrut, Bofors, Sweden (1960), p 15

5) ASTM D1364–64 (Reapproved 1970), "Standard Method of Test for Water in Volatile Solvents" (Fischer Reagent Titration Method)

Following is the description of Method 101.5 of MIL-STD-286B:

1. *Scope*

1.1 This method describes the materials, equipment, and procedures for determining the percentage of moisture in casting solvent, casting powder, casting powder ingredients, and case propellant by the Karl Fischer (KF) method (manual and semi-automatic titrations). This procedure is designed for water contents of less than 1%

1.2 *Limitations and interferences.* The Karl Fischer reagent is composed of pyridine, sulfur dioxide, and iodine dissolved in either methyl alcohol or ethylene glycol monomethyl ether. Substances which react with any of these components will interfere. For example, Karl Fischer reagent will react with aldehydes or ketones in the presence of methyl alcohol. Other examples of interfering substances are oxidizing agents such as peroxides, cupric and ferric salts; reducing agents such as sodium thiosulfate, sodium arsenite, stannous salts and hydrazine; and oxygenated compounds such as metal hydroxides and oxides, bicarbonates and carbonates, and boric acid

2. *Specimen*

2.1 Care must be taken that all samples are protected from gain or loss of water before testing. Propellant samples must be reduced in size to about 1/8-inch cubes to readily dissolve in the solvent. The mode in which this is done is left to the discretion of the installation as it is a function of sample stability and general safety

3. *Apparatus*

3.1 Aquameter (Beckman Instruments, Inc, Model KF2 or KF3 or equivalent. Equivalents are described in ASTM E203-62T)

3.2 Titration Flask, protected from atmosphere with provisions for electrodes and burets

3.3 Magnetic Stirrer

3.4 Buret, 10ml capacity, 0.02ml divisions

4. *Material*

4.1 Karl Fischer Reagent, single solution stabilized, methyl cellosolve as solvent, eg, catalog No So-K-3, diluted to about 2mg/ml titer with methyl cellosolve or a commercial diluent, eg, catalog No So-K-5 of Fisher Scientific Co

4.2 Solvents

Pyridine-Methanol 1:1; mix equal volumes of reagent quality of each solvent containing less than 0.10% water. This must be maintained in a protected system

4.3 Standards

a) Sodium Tartrate Dihydrate, ACS grade containing 15.66% water

b) Water – distilled or equivalent

5. *Calibration and Standardization*

5.1 The water equivalent (mg water/ml KF reagent) is obtained by titrating weighed amts of a standard with the KF reagent as follows:

5.1.1 Add about 50-100ml of the solvent to the titration flask. Maintain a dry nitrogen purge in free volume of container. Using the procedure described in the instrument instruction manual, or the routine procedure for the manual titrating assembly, titrate to a potentiometric end point which remains for 30 seconds. By means of a Lunge pipette, syringe, or equivalent, add one drop of water to the titration flask. Record to the tenth of a milligram the weight of water added, fill the

burette again, and titrate to a 30 second potentiometric end point. Alternately, 200mg of sodium tartrate dihydrate may be added instead of water and may be titrated in like manner. Record the volume of reagent used for the titration

5.1.2 The water equivalent is obtained by dividing the number of milliliters used into the weight of water added. If sodium tartrate dihydrate was added, the calculation is as follows:

Water Equivalent (mg H_2O/mlKF reagent) =

$$\frac{\text{(mg sodium tartrate dihydrate) x 0.1566}}{\text{ml of KF reagent required}}$$

5.1.3 The titer should be about 2mg/ml. If too high or low adjust concentration and restandardize

5.1.4 Frequent standardization is necessary as the reagent is unstable

6. *Procedure*

6.1 Accurately weigh a sample large enough to give a minimum titration of 3ml (5–7ml is desirable)

6.2 Add 50–100ml of the solvent used to the titration flask and titrate to a 30 second end point

6.3 Maintain a dry nitrogen purge over free volume of the flask

6.4 Add the sample, stir until in soln and titrate to a 30 second potentiometric end point

Note: Some samples require about 1 hour to dissolve. Flasks must be well protected to insure water is not absorbed from atmosphere

6.5 *Calculation*

$$\text{Percent water} = \frac{AF}{1000\ W} \times 100$$

where:

A = ml Karl Fischer Reagent used to titrate sample

F = Karl Fischer titer in mg water/ml reagent

W = Sample weight in grams

Ethanol, Ether, Acetone and Water Determinations in Nitrocelluloses and Smokeless Propellants as Described in the Literature:

1) L.A. Chenel, MP **22**, 143–44(1926) & CA **22**, 166(1928) (Determination of water and ethanol in dehydrated NC was carried out at the Laboratoire Central des Poudres by heating a 7–10g sample in a special U-tube in a glycerol bath for 24–30 hours, while the dry air was drawn thru the tube into two tared absorption tubes. The 1st tube contd dry K_2CO_3 pumice to absorb water, while the 2nd contd sulfuric acid pumice to absorb ethanol. After weighing the tubes, the passing of air thru the tube with sample was repeated for 6 hours and the absorption tubes reweighed. If no increase in wt was observed the test was regarded as completed)

2) L.A. Chenel, MP **23**, 4–6(1928) & CA **22**, 3531(1928) (Detn of alcohol, ether and water in solvent eliminated from propellant during drying)

3) D.M. Smith & D.M.D. Bryant, JACS **57**, 61–5 (1935) (Rapid determination of hydroxyl group by means of acetyl chloride and pyridine)

Note: More recent description is given in Ref 6 and in StdMethodsChemAnalysis **2A** (1963), pp 455–57

4) B.E. Christensen et al, IEC (AnalEd) **13**, 821(1941) (Detn of hydroxyl group by esterification with pure acetyl chloride, Problems of volatility and activity of reagent are surmounted)

5) J.W. Peterson et al, IEC (AnalEd) **15**, 225 (1943) (Microdetn of hydroxyl group using acetic anhydride-pyridine in hermetically sealed tube)

6) C.L. Ogg et al, IEC (AnalEd) **17**, 394(1945) (Detn of hydroxyl groups by esterification using acetic anhydride in presence of pyridine)

Note: This method, as well as other methods for detn hydroxyl groups, are described in StdMethodsChemAnalysis **2A**(1963), pp 455–59

7) P.J. Elwing & B. Warskowsky, AnalChem **19**, 1006(1947) (Detn of hydroxyl groups by esterification using phthalic anhydride)

Note: This method is described in StdMethodChemAnalysis **2A**(1963), p 458

8) T.G. Bonner, Analyst **72**, 47–54(1947) [Estimation of Ethanol and Ether in Propellants which Might Also Contain Acetone, and Other Ingredients (such as camphor, diphenylamine, dibutylphthalate, diethylphthalate and diphenylurea)

Procedure:

A. By means of a steel pestle and mortar, crush the propInt sample into pieces of about 3mm across, weigh accurately 2–3g and add it to 25ml of nitrobenzene contd in 250-ml, 3-neck flask. The middle neck of the flask is provided with a gas-tight stirrer, the 2nd neck with a reflux condenser and the 3rd neck with a tube to

bubble the air thru the flask. Connect the upper end of the reflux condenser to three special, spiral absorption tubes connected in series

Notes:

a) The soln in the *1st tube* is intended to oxidize ethanol, while allowing ether, acetone and camphor to pass unchanged. The soln is prepd by mixing in a 100-ml volumetric flask 25ml of aqueous soln contg exactly 8g of $K_2Cr_2O_7$ per liter, plus 2.5ml of concd H_2SO_4. The soln is transferred but not completely to the 1st tube, while the flask is retained without rinsing it, because the soln will be eventually returned to it

b) The soln in the *2nd tube* is intended to remove acetone and camphor which are present in some proplnts. Acetone and camphor being not oxidized in the 1st tube, are oxidized in the stronger soln of the 2nd tube. This soln is prepd by boiling gently in a 2-liter, one-neck flask, under reflux for 10 hours, 104g of $KMnO_4$ and 261g of KOH in one liter of water. After allowing to cool, the supernatant soln is poured off and kept in a stoppered bottle. A 30-ml portion of it is used to fill the 2nd absorption tube

c) The soln in the *3rd tube* serves to oxidize the ether. It is prepd by mixing, with cooling, in a 200-ml volumetric flask 25ml of an aq soln contg exactly 16g of $K_2Cr_2O_7$ per liter with 25ml of concd sulfuric acid. After pouring the bulk of this soln in the 3rd absorption tube, the flask is retained without rinsing, as the soln is eventually returned to it

B. Stir the nitrobenzene soln of sample in 3-neck flask vigorously and with flask immersed in hot water bath, pass a stream of air thru the soln at the rate of 1–2 bubbles per second for 5 hours

C. Stop the flow of air, wash the dichromate solns of tubes 1 and 3 back into their respective volumetric flasks and make up to the marks with water

D. Pipet out 25ml from the 1st flask and add it with cooling and stirring to about 30ml of 10% aq KI soln. After 2 minutes, dilute with water to about 100ml and titrate the liberated iodine with 0.05N of $Na_2S_2O_3$ soln using starch indicator. Calculate the amt of ethanol oxidized in the 1st tube knowing that 1g of $K_2Cr_2O_7$ corresponds to 0.02344g of ethanol

E. The 2nd solution is present to oxidize acetone, camphor, etc which otherwise would get into the 3rd tube, be oxidized, and introduce an error in the ether determination

Note: A determination of acet in proplnts is given by Bonner in Analyst **72**, 434–439 using an extraction method to separate the acetone and an iodine thiosulfate method to measure the acetone

F. Pipet out 25ml from the 3rd flask and add it with stirring and cooling to about 100ml of 10% aq KI soln. After 2 minutes, dilute with water to about 200ml and titrate the liberated iodine with $Na_2S_2O_3$ soln. Calculate the amount of ether, knowing that 1g of $K_2Cr_2O_7$ corresponds to 0.1886g of ether]

9) G. Fleury & H. Liogier, "Dosage du Dissolvant Résiduel dans les Poudres B", MP **30** 223–28 (1948) [Estimation of Ethanol and Ether in Propellants in Absence of Acetone or Ethyl Acetate

Following is the method developed at the Poudrerie Nationale de Saint Médard before WWII but described only in 1948

Briefly, the method is based on the following operations:

a) Saponification of proplnt by Na hydroxide soln

b) Separation of alcohol and ether based on the great difference between their vapor pressures in the neighborhood of RT and on the solubility in water of alcohol and of insolubility of ether

c) Estimation of amounts of separated alcohol and ether by K dichromate in strong sulfuric acid medium

Apparatus, presented in Fig Et 10 consists of the following items:

A – Bubbler, contg concd sulfuric acid (or K dichromate soln in it), serving to count the number of bubbles of air entering thru stopcock R_2 and to retain any gas or vapor which might adversely affect the analysis

B – Round bottom flask, called "un ballon de saponification"

C – Ascending condenser, syst Vigreux (40–45cm long) connected in series with

D – Descending condenser of the same syst and length, which joins at its bottom to a tube provided with three-way stopcock R_3, which can be communicated either with:

E – Volumetric flask of 200ml, or

F – Bubbler, syst Mayer, consisting of 10 bulbs, which joins to:

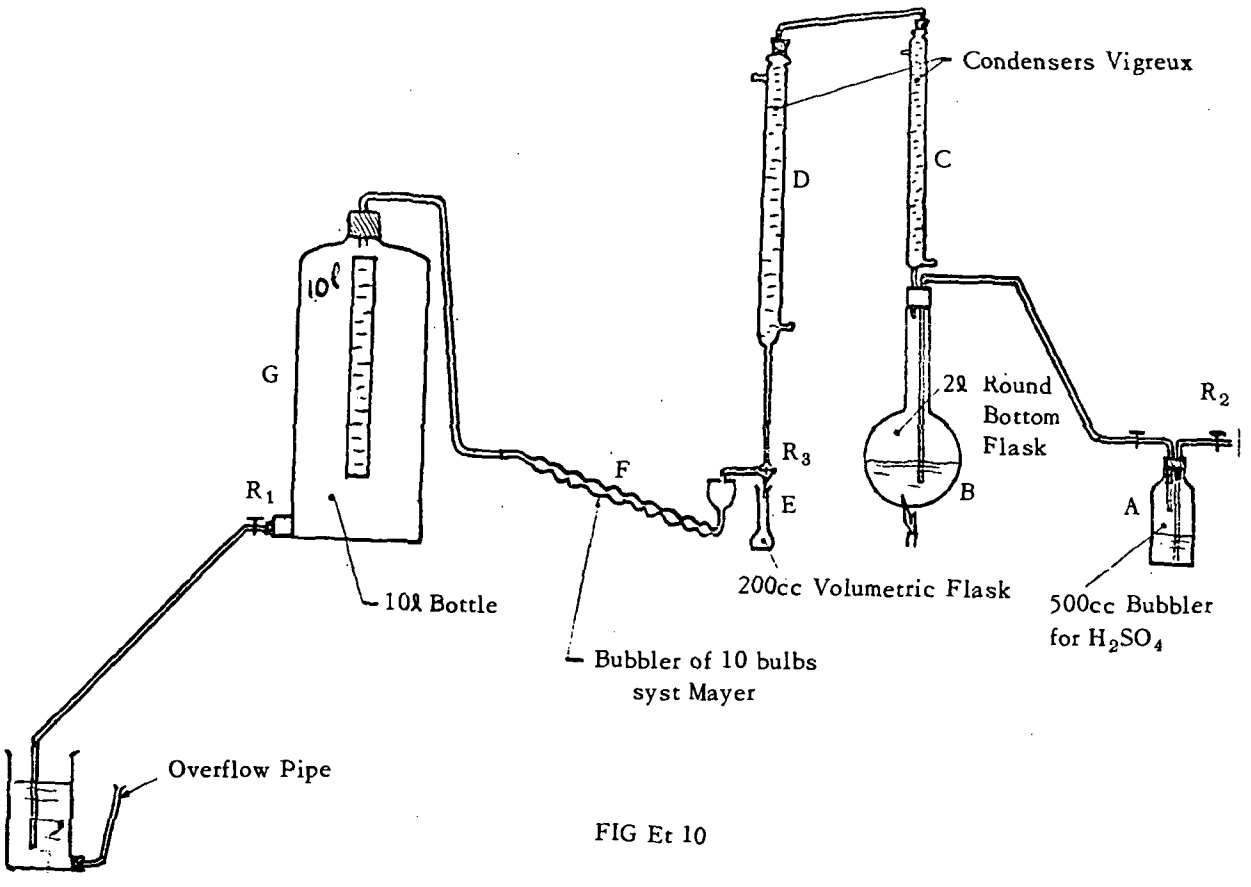


FIG Et 10

G – Bottle of 10 liters, filled with water and connected at the bottom to the stopcock R_1. The water slowly dripping from the bottle during the test permits maintaining in apparatus a slight vacuum of 50–60cm of water, which prevents the escape of alcohol and ether from apparatus during the test

Procedure. It may be subdivided into the following operations:

Saponification: Place 50g sample of proplnt in flask B contg 500ml of aq NaOH soln of 12°Bé (about 8%) and at the same time fill the bubbler F with exactly 50ml of 1.6N K dichromate soln in concd sulfuric acid. Close stopcock R_2, open R_3 to communicate D with F and slightly open R_1 in order to create a small vacuum in the system. After admitting cold tap water to circulate thru the condensers, start to heat the flask B to the boiling point of NaOH soln in order to saponify the proplnt contd in the flask. After saponification is achieved (which usually takes about 1½ hours), continue light boiling in B and start to admit air by opening R_2 to such an extent that about 100 bubbles per minute will go thru A. Continue light boiling and bubbling the air until about 1.51 l of water will be replaced in bottle A by air. This takes from ¾ to 1½ hours of time. During the continued heating and bubbling of air, vapors of alcohol and water will be condensed in C and fall back into the flask B, but vapors of ether will pass thru C and become condensed in the descending condenser D. Then liquid ether will go thru R_3 to bubbler F, where it will be partly oxidized by dichromate soln to form acetic acid

Separation of alcohol. Turn the stopcock R_3 toward volumetric flask E, and stop circulation of cooling water thru condenser C, while continuing to cool the condenser D. This will permit the alcohol to be distilled, pass thru C to D to be condensed in it together with water, into the flask E. After collecting about 150ml of distillate in E, filter it into another 200ml volumetric flask (to remove DPhA) and complete to the mark with distd w

Determination of alcohol. Transfer quantitatively 50ml of previous alcoholic soln into Erlenmeyer flask, incline it at 45° and pour slowly on the side of the flask, with cooling and swirling, 40ml concd sulfuric acid, followed by 30ml of 1.6N K dichromate soln in sulfuric acid. Heat on the steam bath for 20 mins in order to oxidize alc to acetic acid. Cool to RT, add KI soln and titrate the liberated iodine by means of N/10 Na thiosulfate soln using starch indicator

Determination of ether. Transfer one-half (25ml) of dichromate soln from bubbler F into an Erlenmeyer flask, add about 20ml concd sulfuric acid, heat on a steam bath for 20 mins, cool to RT, add KI soln and titrate the liberated iodine by means of N/10 Na thiosulfate soln using starch indicator]

10) J. Tranchant, MP **35**, 305–06(1953) [Determination of alcohol and ether in propellants, in presence of acetone and ethyl acetate combines the method of T.G. Bonner described here as Ref 6 with the method developed at the Poudrerie Nationale de Saint Médard and described by G. Fleury & H. Fleury in MP **30**, 223–28(1948)] (See our translation given as Ref 6)

11) R. Dalbert & J. Tranchant, MP **36**, 189–98 (1954) & CA **49**, 16438(1955) (Study of the determination of the residual solvent in propellants. I. Case of aged powders. II. Case of dinitrotoluene powders)

12) H. Liogier, MP **38**, 167–72(1956) & CA **51**, 11719(1957) (Differential Determination of Residual Alcohol and Ether in Propellants Containing Heavy Esters)

13) J.S. Fritz & G.H. Schenk, AnalChem **31**, 1808(1959) (Detn of hydroxyl group using acetic anhydride in pyridine reagent catalyzed with perchloric acid for esterification)

Note: This method is described in StdMethodsChemAnalysis **2A**, pp 456–57

14) S. Siggia & G.J. Hanna, AnalChem **33**, 900–01(1961) (Detn of hydroxyl groups using pyromellitic dianhydride for esterification)

Note: This method is described in StdMethodsChemAnalysis **2A**(1963), pp 458–59

Ethanol, Ether, Acetone and Water Determinations in Solvents Used in Manufacture of Smokeless Propellants, as Described in the Literature:

1) M. Marqueyrol & E. Goutal, MP **19**, 368–80 (1922) & CA **17**, 3656(1923) (Analysis of mixtures of alcohol, ether and water can be made by determining the density of the sample in its original condition and after the addition of measured quantity of water until the liquid becomes turbid. Enclosed in the paper are triaxial diagrams, which help to make necessary calculations)

2) W.M. Fischer & A. Schmidt, Ber **57**, 693–98 (1924) & **59**, 679–82(1926) (Determination of ethanol or methanol by means of Na nitrite, followed by removal of ethyl-, or methylnitrite, with a current of carbon dioxide, followed by absorption of gas in concd Na bicarbonate soln. On action of KI soln in dilute HCl on the soln contg nitrites, the iodine is liberated and then the free iodine is detd by titration with Na thiosulfate, $Na_2S_2O_3$)

3) N.V. Chalov & L.P. Vol'skaya, Zavodskaya-Lab **12**, 286–91(1946) & CA **41**, 3399–4000 (1947) (Detn of ethanol in dilute aqueous solns by a method, which is modification of the method of Fischer & Schmidt, listed here as Ref 2. Both methods are based on the conversion of alcohol to its nitrite and total time required is 20 mins. Sensitivity is as low as 0.025–0.005% and accuracy ±3%. A fairly detailed description of the method is given in the above CA)

4) N.V. Chalov, RussP 69874(1947) & CA **44**, 78(1950) (Description of the same method as given in previous Ref 3)

5) J. Lamond, Analyst **74**, 560–61(1949) [Small quantities of alcohol in ether may be detd after extracting it from ether by water, followed by testing the aqueous extract by means of ceric ammonium nitrate. The following reaction takes place, producing the red coordination product:

$$C_2H_5OH+(NH_4)_2Ce(NO_3)_6 \rightarrow CeOC_2H_5(NO_3)_5\cdot(NH_4)_2+HNO_3$$

The intensity of red coloration produced by this reaction, which may be measured by means of a colorimeter is proportional to the amount of alcohol in ether]

6) V.W. Reid & R.K. Truelove, Analyst **77**, 325 (1952) (Colorimetric method for determining trace quantities of hydroxy-compounds by means of ceric ammonium nitrate) [See description of this method in StdMethodsChemAnalysis **2A** (1963), p 462]

7) G. Bourjol, MP **35**, 197–98(1953) (Rapid method of analysis of ethanol-ether-water mix-

tures by combining density measurements with Karl Fischer's method for determination of water. Knowing these values, the contents of alcohol and in ether in the mixture may be read directly from a specially prepd triangular diagram. This method is modification of Marqueyrol & Goutal's method described here as Ref 1. In his paper Bourjol reviewed the method of analysis of triple mixture consisting of distillation using fractionating column in order to separate the ingredients into two fractions: ether-alcohol and alcohol-water, followed by determination of density for each fraction)

Ethanol Azido-, Nitroso- and Nitro-Derivatives, Ethanolazide,2-(or β)-Azidoethanol,2-Azidoethyl Alcohol or 2-Triazo-1-ethanol, $N_3.CH_2.CH_2.OH$; mw 87.08, N 48.26%, OB to CO_2 −101%; col liq, sp gr 1.1435 at 24.9°, fr p (?), bp 60° at 8mm & 73° at 20mm; explodes when dropped on a hot plate. Its vapors are odorless, but highly toxic. It was first prepd in 1867 by Butlerov & Ossokin (Refs 1 & 2) and later by Forster & Fierz (Ref 3) by heating on a water bath for 48 hrs a mixture of equal parts of ethylenechlorohydrine and Na azide. It was also prepd and investigated by Urbański (Ref 4) and A.D. Little (Ref 6). Blatt (Ref 5) stated that it is an explosive about one-half as powerful as TNT, insensitive to impact and unstable in storage. (See also Refs 6, 7, 8 & 9)

Refs: 1) Beil **1**, 340 & (171) 2) Butlerov & Ossokin, Ann **144**, 40(1867) 3) M. Forster & H. Fierz, JCS **93**, 1866(1908) 4) T. Urbański, IX CongrInternlQuímPuraAplicada **4**, 438(1934) & CA **30**, 3649(1936) 5) A.H. Blatt, OSRD **2014**(1944) (Azidoethyl Alcohol) 6) ADL, PureExplCompds, Part **1**(1947), p 48 (Trauzl Test value 50% PA or 55% TNT) 7) H.O. Spauschuss & J.M. Scott, JACS **73**, 208–12(1951) & CA **45**, 7002(1951) (Molar refractions and parachlors) 8) T.F. Fagley, E. Klein & J.F. Albrecht, Jr, JACS **74**, 3104–6 (1952) & CA **47**, 9748(1953) (Heat of Combustion) 9) P. Gray & T.C. Waddington, Proc-RoySoc **325A**, 481–95(1956) & CA **50**, 15203 (1956) (Dissociation energy of $C-N_3$ bond)

Ethanol Azidonitrate; 2-Azido-1-ethanol Nitrate; β-Azidoethylalcohol Nitrate or **2-Triazo-1-ethanol Nitrate,** $N_3.CH_2.CH_2.ONO_2$; mw 132.08, N 42.42%, OB to CO_2 −36.3%; volatile, toxic liq, sp gr 1.34, fr p −20°, bp – explodes at 180° when heated at the rate of 25°C/min; gelatinizes NC; resembles NGc in its physiological and explosive properties. It was prepd by Urbański (Ref 4) by nitrating ethanol azide with mixed nitric-sulfuric acid. It seems that it can be prepd by treating nitrochlorohydrin (obtd by nitrating chlorohydrin) with Na azide. Azidonitrate was also prepd by Bergeim (Ref 2) who patented its use in dynamite-type expl mixtures, such as contg Azidoethanol Nitrate, NG, NGc, Na nitrate, woodmeal and Ca carbonate. Fishbein (Ref 3) reviewed its manuf and properties. A.D. Little, Inc (Ref 7) prepd it and detd some of its props. The compd was listed in Ref 5

The following explosive props are given in Urbański (Ref 4), Blatt (Ref 6) & ADL (Ref 7)
Impact Sensitivity – much more sensitive than NG or NGc; when absorbed on filter paper the pure liquid can be detonated with a hammer. For a 50/50 mixt with sawdust the max fall for no shots with 10-lb wt was 2 inches, while corresponding value for NGc was 5 and for NG 6 inches
Power, by Ballistic Mortar – 141% TNT
Power, by Trauzl Test – 150% TNT (and 171% PA?)
Rate of Detonation – 1985 and 6550m/sec at sp gr 1.34
Refs: 1) Beil **1**, {1365} 2) F.H. Bergeim, USP 1620715(1927) & CA **21**, 1551(1927) 3) M.S. Fishbein, VoyennayaKhimiya(Russia) **1933**, No 6, pp 3–8 & CA **29**, 7077(1935) 4) T. Urbański, IX CongrInternlQuimPuraAplicada **4**, 438–46 (1934) & CA **30**, 3649(1936) 5) Clift & Fedoroff **2**(1943), p E12 6) A.H. Blatt, OSRD **2014**(1944) (Azidoethyl Nitrate) 7) ADL, PureExplCompds, Part **1**(1947), p 48

Ethanol,2-Nitro; 2-Nitroethanol-1; 1-Hydroxy-2-nitroethane; or **β-Nitroethylalcohol,** $O_2N.CH_2.CH_2.OH$; mw 91.07, N 15.38%, OB to CO_2 −61.5%; colorless liquid; sp gr 1.296 at 20/20°, n_D 1.4447 at 20°, fr p <−80°, bp 194° at 765mm, 119–20° at 35mm & 101° at 15mm; chars without expln at 260°; sol in w, alc & eth. It can be prepd in impure state by treating 1 mole ethylene with 1 mole of white concd nitric acid by the method of Wieland & Sakellarios(Refs 1, 2 & 3). The method of

Gorski & Makarov (Ref 4) consists of treating Nitromethane with paraformaldehyde in the presence of K carbonate. Yields are 43–45%. Pure compd can be prepd by treating an ethereal soln of $CH_2I.CH_2OH$ with Ag nitrate (See also Ref 5). Its heats of combstn & formation are given by Médard & Thomas (Ref 9).

Blatt (Ref 6) and ADL Rept (Ref 7) list the following props:

Abel Heat Test at 82.2° – 3 mins (NG 10 mins)
Friction Sensitivity – very insensitive
Impact Sensitivity – very insensitive
Initiation by Detonators – incomplete with No 8 Briska Detonator in Trauzl Test
Power by Ballistic Mortar – 62% Blasting Gelatin, which corresponds to about 110% TNT
Power by Trauzl Test – 106% TNT
Thermal Stability at 120° – no brown fumes after 5 hours
Vacuum Stability – 3.85cc in 40 hrs

Due to the fact that it easily gelatinizes NC forming a thick homogeneous mass, it could be recommended as an expl plasticizer for NC in prepn of smokeless proplnts, if its stability were more satisfactory

It was proposed for use as an explosive by Levy & Scaife (Ref 6a)

2-Nitroethanol forms a *Potassium Derivative*, K *aci*-nitroacetaldehyde, $K^+(OCH{:}CH.NO_2)^-$, mp 150–60° (decompn) (Ref 8)

Refs: 1) Beil **1**, 339–40, (171) & [339] 1a) R. Demuth & V. Meyer, Ann **256**, 29 (1890) 2) H. Wieland & E. Sakellarios, Ber **53**, 207–08 (1920) 3) Naoúm, NG (1928), 220 4) M. Gorski & S.P. Makarov, Ber **67**, 996 (1934) 5) J.R. Johnson, "Preparation of Nitrocompounds", OSRD **98** (1941), 19–21 6) Blatt, OSRD **2014** (1944) 6a) N. Levy & C.W. Scaife, JCS **1946**, 1093 & CA **41**, 2388 (1947) 7) ADL, PureExpl-Compds, Part 1 (1947), p 84 8) C.D. Hurd & L. Sherwood, JOC **12**, 471–76 (1948) & CA **43**, 1004 (1949) 9) L. Médard & M. Thomas, MP **35**, 155–73 (1953) (Heat of combstn at const vol 275.8kcal/mole) 10) V.I. Burmistrov & V.M. Bashinova, NeftepererabNeftekhim **1**, 21–3 (1968) (Russ) & CA **68**, 104503 (1968) (Phys props of 2-Nitroethanol) 11) Ibid, ZhPriklKhim **41** (8), 1853–58 (1968) & CA **69**, 105798 (1968) (Prepn) 12) H.N. Lee & R.W. Van House, USP 3426084 (1969) & CA **70**, 67594 (1969) (Manuf)
Part 1 (1947), p 84 8) C.D. Hurd & L.T. Sherwood, JOC **13**, 471–6 (1948) & CA **43**, 1004b (1949) 9) L. Médard & M. Thomas, MP **35**, 156 & 172 (1953)

Ethanol,2-Nitroso; 2-Nitrosoethan-1-ol; 1-Hydroxy-2-nitrosoethane or *β-Nitroso-ethylalcohol*, $ON.CH_2.CH_2.OH$. Not found in the literature thru 1966

Ethanol,2-Nitronitrate; 2-Nitroethanol Nitrate; 1-Nitroxy-2-nitroethane or **β-Nitroethyl Nitrate**, $O_2N.CH_2.CH_2.ONO_2$; mw 136.07, N 20.59%, OB to CO_2 –11.7%; very volatile, toxic liquid with irritating odor; sp gr 1.468 at 18°, mp – not given, bp 120–122° at 17mm and 103° at 15mm; decomp at 185° with evoln of brown fumes and chars at 260°; sol in w; distills with vapors of w & alc; being sl acidic, it dissolves in cold caustic alkali solns, giving a yellow color. It was first prepd in 1920 by Wieland & Sakellarios (Refs 1 & 3) from the same kind of oil as was obtd by Kekulé in 1869 (Refs 1 & 2) on passing ethylene into a mixture of concd nitric and sulfuric acid. On distilling Kekulé oil in steam and in vacuum were obtd Nitroglycol, bp 105° at 19mm and β-Nitroethyl Nitrate, bp 120–122° at 17mm

The action of mixed acid on ethylene can be represented as follows:

$$\begin{matrix} CH_2 \\ \| \\ CH_2 \end{matrix} + HNO_3 \rightarrow \begin{matrix} CH_2.NO_2 \\ | \\ CH_2OH \end{matrix} + HNO_3 \rightarrow$$

Nitroethanol

$$\rightarrow \begin{matrix} CH_2.NO_2 \\ | \\ CH_2.ONO_2 \end{matrix} + H_2O$$

Nitroethanol Nitrate

which proceeds simultaneously with formation of Ethyleneglycol Dinitrate:

$$\begin{matrix} CH_2 \\ \| \\ CH_2 \end{matrix} + 3HNO_3 \rightarrow \begin{matrix} CH_2.ONO_2 \\ | \\ CH_2.ONO_2 \end{matrix} + HNO_2 + H_2O$$

The crude Kekulé oil, when freed from admixed and dissolved acid by washing with water, contd 40–50% 2-Nitroethanol Nitrate and 50–40% Nitroglycol. On shaking the oil with very dil caustic alkali, the Nitroethanol Nitrate is removed leaving pure Nitroglycol. Numerous patents have been issued for processes of procuring pure Nitroglycol from Kekulé oil (Refs 4 & 5)

2-Nitroethanol Nitrate was also obtd by the direct action of mixed acid on 2-Nitroethanol (Ref 4, p 220)

Nitroethanol Nitrate is an explosive compd,

comparable in power to NG but very insensitive to friction and impact, very volatile and of unsatisfactory stability. It easily gelatinizes Collodion Cotton

Following properties are listed in Blatt (Ref 6) and in ADL (Ref 7):

Abel Heat Test at 82.2° – 30 seconds, vs 10 mins for NG
Hygroscopicity – none
Impact Test with 10kg wt – positive at 110cm fall
Power, by Ballistic Mortar Test – 102% NG
Power, by Trauzl Test – 173% TNT

Note: A Blasting Gelatin contg 93% of 2-Nitroethanol Nitrate and 7% Collodion Cotton produced a lead block expansion (Trauzl Test) of 500cc vs 600cc for Blasting Gelatin contg 93% NG. The same two expl gelatins tested by Ballistic Mortar gave a slightly higher value for gelatin contg 2-Nitroethanol Nitrate

Thermal Stability at 120° – brown fumes after 90 mins
Vacuum Stability at 100° – 11.5cc/40 hours

Its use in expls is not recommended on account of high volatility and unsatisfactory stability

It is described as an explosive in Ref 8 and its *Potassium Salt*, $C_2H_3KN_2O_5$ is described in Ref 9

Refs: 1) Beil **1**, [339–40] 2) F.A. Kekulé, Ber **2**, 329(1869) 3) H. Wieland & E. Sakellarios, Ber **53**, 201(1920) 4) Naoúm, NG (1928), 220–21 5) Davis (1943), 228 6) A.H. Blatt, OSRD **2014**(1944) (Nitroethyl Nitrate) 7) ADL, PureExplCompds, Part **1** (1947), p 119 8) N. Levy & C.W. Scaife, JCS **1946**, 1093–6 & CA **41**, 2388(1947) 9) K. Klager, USP 2640072(1953) & CA **48**, 7626(1954)

Ethanol, 2-Nitronitrite; 2-Nitroethanol Nitrite; 1-Nitrosoxy-2-nitroethanol or *β-Nitroethylalcohol Nitrite*, $O_2N.CH_2.CH_2.ONO$; mw 120.065, N 23.33%, OB to CO_2 –26.6%; not found in the open literature

Refs: 1) Beil, not found 2) CA, not found

Ethanol-2,2-Dinitro; 2,2-Dinitroethanol, 1-Hydroxy-2,2-dinitroethanol or **β-Dinitroethyl Alcohol**, $(O_2N)_2CH.CH_2.OH$; mw 136.06, N 20.58%, OB to CO_2 –11.7%; colorless oil; mp 2–3°(wh crysts from ethyl chloride); bp 50–60°(0.2mm Hg, cyclic molecular still); n_D 1.471 at 20°, stable at –20°; sol in w, eth; corrosive to skin; prepd by acidification with sulfuric acid of the potas- sol in w, eth; corrosive to skin; prepd by acidification with sulfuric acid of the potassium salt of the *aci*-form prepd from formaldehyde and potassium dinitromethanate (Ref 2)

Refs: 1) Beil **1**, 340 2) P. Duden & G. Pinndorf, Ber **38**, 2033(1905) 3) H. Feuer, G.B. Bachman & W. May, JACS **76**, 5124–6 (1954) & CA **49**, 13091i(1955) (Sodium salt, mp 132°, expl 135–6° 4) M.H. Gold, E.E. Hamel & K. Klager, JOC **22**, 1665–7(1957) & CA **52**, 8936c(1958) (Prepn) 5) M.J. Kamlet & D.J. Glover, JOC **27**, 537–43(1962) & CA **57**, 2050g(1962) (UV spectrum of K-salt) 6) M.J. Kamlet, R.E. Oesterling & H.G. Adolph, JChemSoc **1965**, 5838–49 & CA **64**, 1487e (1966) (IR spectrum of K-salt)

Ethanol-2,2,2-trinitro; 2,2,2-Trinitroethan-1-ol; 1-Hydroxy-2,2,2-trinitroethane or **β-Trinitroethyl Alcohol**, $(O_2N)_3C.CH_2.OH$; mw 181.06, N 23.21%, OB to CO_2 +13.2%; colorless hygroscopic ndls; mp 27–30°(Ref 4) or 36–37°(Ref 6) or 72° (Ref 9); bp 103° at 14mm and 77–80° at 4mm; n_D 1.4578 at 20°; sl sol in cold w and very sol in hot w. It was first prepd in 1941 by C.H. Hurd & A.C. Starke of Northwestern University, but their method was not published in open literature: it consisted of interaction between nitroform and paraformaldehyde:

$$3CH(NO_2)_3 + (HCHO)_3 \rightarrow (O_2N)_3C.CH_2OH$$

The same method, essentially, was used by later investigators (Refs 2 & 4) and also by Ficheroulle & Gay-Lussac (Ref 6). They treated nitroform by slowly adding an excess of paraformaldehyde (which they called trioxymethylène), keeping the mixture at below 70–80° by cooling because the reaction is very exothermic. The resulting trinitroethanol can be distd betw 80 & 93° at 5mm pressure and crystallized in colorless ndls of mp 36–37°. Yield 65%. Distn is very hazardous and for this reason careful control of temp is very important. Purification by forming the acetate, distg it betw 85 & 95°(in vacuo), followed by saponification is recommended by F & G(Ref 6). US Rubber Co (Ref 3) prepd it from nitroform,

formaldehyde & KOH, obtg crysts (after purification) with bp 80° at 2mm pressure and detonating by a hammer blow. Schenck & Wetterholm (Ref 7) prepd it and used it as a starting material for prepn of bis(2,2,2-trinitroethylene)-diamine

Its heat of combstn was detd at PicArsn as 1291kcal/kg

Accdg to Ficheroulle & Gay-Lussac (Ref 6), Trinitroethanol is unstable and even at RT evolves nitrous fumes. When heated to 120° it rapidly decomposes with evolution of nitrous fumes and then violently explodes. Its sensitivity to shock is comparable to that of NG. Although it is a good gelatinizer for NC, it cannot be recommended for use in expls and propInts on account of its poor stability. In the 100°C Heat Test, it lost 68% 1st 48 hrs and 26% second 4 hrs, with no expln in 100 hrs

Refs: 1) Beil, not found 2) W. Hunter et al, BIOS Final Rept No **709**, HMStationary Office, London (1946), p 9 (Description of prepn of Trinitroethanol by Dr Schimmelschmidt of IG Farbenindustrie, Höchst-am-Main) 3) US Rubber Co Rept **No 4**(1948), p 9 and **No 5**(1948–49), p 20 (Prepn and props of Trinitroethanol) 4) N.S. Marans & R.P. Zelinski, JACS **72**, 5329–30(1950) & CA **45**, 4642(1951) (Prepn and props of Trinitroethanol) 5) ADL, Synthesis HE's, 2nd Rept (1951), p 33 (Prepn & props of Trinitroethanol) 6) H. Ficheroulle & A. Gay-Lussac, MP **34**, 121–23 (1952) & CA **48**, 4838–39(1954) 7) F.R. Schenck & G.A. Wetterholm, SwedP 148217 (1954); Ibid, BritP 729469(1955) & Ibid, USP 2731460(1956); CA **50**, 1893 & 7125(1956) (Prepn & props) 8) J. Ville, MP **42**, 22 & 24 (1960) & CA **55**, 18109(1961) (Prepn & props of Trinitroethanol) 9) H. Feuer & T.J. Kucera, JOC **25**, 2069(1960) & CA **55**, 14286e (1961) (Prepn) 10) H.E. Ungnade & L.W. Kissinger, JOC **31**(2), 369(1966) & CA **64**, 11081d(1966) (IR, UV spectra) 11) N.D. Lebedeva, V.L. Ryadnenko & I.N. Kuznetsova, ZhFizKhim **42**(7), 1627(1968) & CA **69**, 90540c(1968) (Props)

Ethanol-2,2,2-trinitro-1-nitrate; 1-Nitroxy-2,2,2-trinitroethane; 2,2,2-Trinitroethyl Alcohol Nitrate, $(O_2N)_3C.CH_2(ONO_2)$; mw 226.07, N 24.78%, OB to CO_2 +28.3%. This compd was considered to be too sensitive for use as an HE (Ref 2)

Preparation of this compd is probably by nitration of trinitroethanol prepared from nitroform and formaldehyde

Refs: 1) Beil, not found 2) CA, not found 3) A.H. Blatt, OSRD Rept **2014**(1944) (2,2,2-Trinitroethyl Nitrate) 4) ADL, PureExplCompds, Par **1**(1947), p 120

Ethanol Acetate and Derivatives. See Ethyl Acetate and Derivatives

Ethanolamine and Derivatives. See AMINOETHANOL (ETHANOLAMINE) AND DERIVATIVES in Vol 1 of Encycl, pp A200 & A201 and the following Addnl Refs:

A) P. Varrato, PATR **104**(1931) (Study of nitration of ethanolamine and properties of products of nitration)

B) A. Foulon, SS **27**, 399(1932) & MAF **14**, 462(1935)(Description of prepn and props of Ethanolamine Dinitrate. It is considered unsuitable as a military expl on account of its hygroscopicity and instability)

C) L. Médard & M. Thomas, MP **38**, 45–63 (1956) & CA **51**, 13553g(1957) (Heat of combustion of $O_2NO.CH_2.CH_2.NH_2.HNO_3$ given as 1875.2cal/g at 18°)

D) I. Lafontaine, BullBelg **67**, 153–66(1958) & CA **53**, 791d(1959) (Dielectric constant of parent at 20°, 25°)

E) M. Kuhn, W. Luttke & R. Mecke, ZAnalChem **170**, 106–14(1959) & CA **54**, 2938h(1960)(IR spectrum of parent)

F) R.A. McDonald, S.A. Shrader & D.R. Stull, JChemEngData **4**, 311–13(1959) & CA **54**, 9712c(1960)(Freezing point of parent given as 10.31°)

G) J. Timmermans & Mme Hennaut-Roland, JChimPhys **56**, 984–1023(1959) & CA **55**, 24g (1961)(Physical constants for parent)

H) G. Werner & K. Keller, Ber **93**, 1982–8 (1960) & CA **55**, 2633c(1961)(Prepn and IR spectrum of parent-$HClO_4$)

I) B.J.R. Nicolaus & E. Testa, AngChem **73**, 655(1961) & CA **56**, 7102f(1962)(Mentions parent-Picrate)

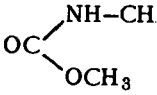
J) J.F. Coetzee, G.R. Padmanabhan & G.P. Cunningham, Talanta **11**(2), 93–103(1964) & CA **60**, 4859a(1964)(Mentions parent-$HClO_4$ and parent-Picrate)

K) H.E. Stokinger et al, JOccupational-

Med **5**(10), 491–8(1963) & CA **60**, 6120f (1964)(Threshold limit value for parent)
L) H. Mueller, GerP 1159731(1963) & CA **60**, 6553h(1964)(Mentions prepn of parent-HN_3)
M) M. Kondo, T. Kuramoto & T. Sato, JapP **70**, 21568(1970) & CA **74**, 56584p(1971)(Mentions an ethanolamine nitrite)
N) G.L. Ryzhova & G.N. Mishustina, TrTomskGosUniv **192**, 182–87(1968)(Russ) & CA **74**, 148569r(1971)(Mentions parent-Trinitrobenzene and parent-Tetranitromethane complexes)

Ethanolamine-Diphenylcarbamide Complexes. See under Diphenylcarbamide Complexes Suitable as Stabilizers and Gelatinizers for Smokeless Propellants, in Vol 5, pp D1445–46

Ethanolaminonitrate-methylurethane,



mw 164.12, N 17.07%, OB to CO_2 –68.3%, bp 115–20°(0.5 torr), sp gr 1.461, n_D 1.4735 at 25°, heat of combustion 2454.9cal/g. Prepn not given in open literature
Refs: 1) Beil, not found 2) P. Tavernier & M. Lamouroux, MP **37**, 201 & 206(1955) & CA **51**, 717a(1957)

Ethanolamino-tetrazido-copper or **Aminoethanol-bis[copper(II) diazide],**
$H_2N.CH_2.CH_2.OH.2Cu(N_3)_2$; mw 292.71, N 62.21%, OB to CO_2 –46.4%, expl 186°(impure, Ref 2). It forms an HCl salt which neither melts nor explodes (Ref 3). See the CA for Ref 2 for prepn
Refs: 1) Beil – not found 2) M. Straumanis & A. Cirulis, ZAnorgChem **251**, 335–54 (1943) & CA **37**, 6574(1943) 3) Ibid, **252**, 9–23(1943) & CA **38**, 3564(1944)

Ethanolaniline and Derivatives. See ANILINOETHANOL AND DERIVATIVES in Vol 1 of Encycl, pp A424 to A429. Its expl derivative 2-(2′,4′,6′-Trinitro-N-nitranilino)-ethanol Nitrate, described on p A425-L, is called β-(2,4,6-Trinitrophenylnitramino)-ethyl Nitrate in Blatt, OSRD **2014**(1944) and code named Pentryl. Compare with **Ethyltetryl**, which is described under N-Ethylaniline or N-Ethylphenylamine and Derivatives

Ethanolbenzene or **Phenylethanol and Derivatives** *Ethanolbenzene, Hydroxyethylbenzene, β-Phenylethyl Alcohol, Phenetyl Alcohol* or *Benzylcarbinol,* $C_6H_5.CH_2.CH_2.OH$; mw 122.16, colorless oil, sp gr 1.023 at 13/4°, n_D^{20} 1.5310, fr p –27°, bp 219–21° at 750mm; sl sol in w; miscible with alc & eth; sol in 50% alc and in glycerol; sl sol in mineral oil. It can be prepd by reduction of phenylacetic ethyl ester by Na in abs alc or by the action of ethylene oxide on phenyl MgBr and subsequent hydrolysis; used in organic synthesis, cosmetics, medicine, etc
Refs: 1) Beil **6**, 478 & [448] 2) CondChemDict (1961), 874-L (Phenetyl Alcohol)

Ethanol-2,4-dinitrobenzene or **β-(2,4-Dinitrophenyl) ethanol,**
$HOCH_2.CH_2.C_6H_3(NO_2)_2$; mw 212.16, N 13.20%. Prepn of the 3,5-dinitro by diborane reduction of $HO_2C.CH_2.C_6H_3$-3,5-$(NO_2)_2$ is given in Ref 3; no props mentioned. Ref 4 mentions prepn by nitration of the parent, but CA does not indicate which isomer. The 2,4-dinitro is mentioned in Refs 2 & 5 without prepn or props
Refs: 1) Beil – not found 2) Y. Ogata & M. Okano, ReptsInstChemRes, Kyoto Univ **17**, 133–6(1949) & CA **46**, 1487e(1952) 3) B.C.S. Rao & G.P. Thakar, CurrentSci(India) **29**, 389 (1960) & CA **55**, 9362g(1961) 4) S.T. Rashevskaya, E.S. Kashcheeva & E.I. Mostoslavskaya, ZhObshchKhim **33**(12), 3998–4002(1963) & CA **60**, 9178f(1964) 5) V.G. Sinyavskii & R.A. Kornienko, ZhOrgKhim **6**(6), 122–23(1970) (Russ) & CA **73**, 66161s(1970)

Ethanol-2,4,6-trinitrobenzene or **β-(2,4,6-Trinitrophenyl)-ethanol,**
$HOCH_2.CH_2.C_6H_2(NO_2)_3$; mw 257.16, N 16.35%, OB to CO_2 –77.8%, mp 112°(Ref 6). It was prepd by refluxing TNT with alkaline HCHO at 90°(Ref 6); used to prepare the corresponding ethyl chloride, mp 72°(Ref 3), 78°(Ref 6); used to prepare the 2,6-dinitro-4-amino compd; yel, mp 161–65°(Ref 2)
Refs: 1) Beil – not found 2) G.D. Parkes & A.C. Farthing, JCS **1948**, 1275–8 & CA **43**, 592g(1948) 3) F. Challenger & P.H. Clapham, JCS **1948**, 1612–15 & CA **43**, 1733g (1949) 4) Z. Pelchowicz & E.D. Bergmann, BullResCouncilIsrael **1**, 134(1951) & CA **46**, 2889d(1952)(Mentions compound and esters)

5) C.F. Bjork, W.A. Gey, J.H. Robson & R.W. Van Dolah, JACS **75**, 1988–9(1953) & CA **49**, 5338i(1955) 6) Z. Bonecki & T. Urbański, BullAcadPolonSci, SerSciChim **9**(7), 463–6 (1961)(Eng) & CA **60**, 2807h(1964)

Ethanolnitrate-nitrobenzene or **β-(Nitrophenyl)-ethanol Nitrate,** $O_2NO.CH_2.CH_2.C_6H_4(NO_2)$; mw 212.6, N 13.20%. Prepn of the ortho compd by nitration of the parent at or below 0°(Refs 2, 3 & 7) by nitrating benzocyclobutene (Refs 5 & 6); bp given as 85–105°(0.05 torr) and n_D 1.546–550 (Ref 2), and 120–40°(2 torr) and n_D^{15} 1.5799–1.5779 (Ref 5). Prepn of the para compd by nitration of parent below 0°(Refs 3 & 7), and nitration of benzocyclobutene (Ref 6); mp given as 56–58°(Ref 3), 32° (Ref 6); n_D^{20}1.5735 (Ref 6). Prepn of meta compd mentioned in Ref 3 by nitration of parent below 0°

Refs: 1) Beil – not found 2) L. Horner, H.G. Schmelzer & B. Thompson, Ber **93**, 1774–81(1960) & CA **54**, 24583e(1960) 3) P.M. Kochergin & L.S. Blinova, USSR 128011(1960) & CA **55**, 3524c(1961) 4) S.W. Tinsley & J.T. Fitzpatrick, USP 3037057(1962) & CA **57**, 12382f(1962) (Prepn of unspecified isomers by nitration of parent below 0°) 5) J.B.F. Lloyd & P.A. Ongley, Tetrahedron **20**(10), 2185–94(1964) & CA **62**, 495a(1965) 6) L. Horner, P.V. Subramaniam & K. Eiben, TetrahedronLetters **1965**(4), 247–50 & CA **62**, 9075f(1965) 7) V.G. Sinyavskii & V.F. Kovaleva, ZhOrgKhim **6**(8), 1692–96(1970) & CA **73**, 98524d(1970)

Ethanol-2,4-dinitrobenzene Nitrate or **β-(2,4-Dinitrophenyl)ethanol Nitrate**

$O_2N.O.CH_2.CH_2.C_6H_3(NO_2)_2$; mw 257.16, N 16.35%, OB to CO_2 –77.8%. Prepn of 2,4- and 2,6-compds by nitration of the parent (Ref 3). Ref 2 indicates an (unspecified) isomer which is insol in water and will gelatinize NC

Refs: 1) Beil – not found 2) G. Knoffler, GerP 1056989(1959) & CA **55**, 6869a(1961) 3) V.G. Sinyavskii & V.F. Kovaleva, ZhOrgKhim **6**(8), 1092–96(1970) & CA **73**, 98524(1970)

Ethanol-2,4,6-trinitrobenzene Nitrate or **β-(2,4,6-Trinitrophenyl)ethanol Nitrate,** called in Blatt: β-(2,4,6-Trinitrophenyl)-ethyl Nitrate, $O_2NO.CH_2.CH_2.C_6H_4(NO_2)_3$, mw 302.16, N 18.54%, OB to CO_2 –53%; solid, mp 83°. It was prepd by Vender in impure state, as oil, by nitrating β-phenylethyl alcohol. Another method was by condensing TNT & formaldehyde to β-(2,4,6-Trinitrophenyl) - ethyl alcohol and nitrating this compd (Ref 2)

The oil product was examined during WWII in the labs of duPont Co and found that it cannot be detonated by the impact of an 8-oz steel ball dropped 25 inches, but was detonated by a hammer blow (Ref 3)

Refs: 1) Beil **6**, (239) 2) V. Vender, Gazz **45**(II), 97(1915) & CA **10**, 1513(1916) 3) A.H. Blatt, OSRD Rept **2014**(1944) 4) Not found in later refs thru 1972

Ethanol Chloride. See Chloroethanol in Vol 3, p C254-R

Ethanol-N-diphenylamine. See Diphenylamino-ethanol in Vol 5, p D1441-R

Ethanol Diphenylurea [1,1-Diphenyl-3-(2-hydroxyethyl)-Urea; 3-(2-hydroxyethyl)-1,1-diphenylurea; and 2-(2-hydroxyethyl)-1,1-diphenylurea (CA)], $(C_6H_5)_2NCONH.CH_2.CH_2OH$; mw 256.25, mp 117.5°, heat of combustion 7349.5cal/g. It was prepd by reacting N,N-diphenylcarbamoyl chloride with ethanolamine (Ref 3)

Refs: 1) Beil – not found 2) P. Tavernier & M. Lamouroux, MP **37**, 203, 206(1955) & CA **51**, 717b(1957) 3) D.E. Rivett & J.F.K. Wilshire, AustralianJChem **19**(1), 165–8(1966) & CA **64**, 19594b(1966)

Ethanolethylenediamine and Derivatives
N-Ethanol;ethylenediamine; N-(2-Hydroxyethyl)-ethylenediamine; or *1-Hydroxy-3,6-diazahexane,* $HO.CH_2.CH_2.NH.CH_2.CH_2.NH_2$; mw 104.15, bp 238–40°, sol in w, alc; hygroscopic; prepd from ethylene oxide and ethylene diamine (Ref 1)

1-Nitroxy-3,6-diazahexane Dinitrate, $O_2N.O.CH_2.CH_2.NH.HNO_3.CH_2.CH_2NH.HNO_3$;

mw 275.18, N 15.27% (NO_2), OB to CO_2 −32.0%, UV spectrum (Ref 2). It was probably prepd by nitration of the parent; nothing in the open literature

Refs: 1) Beil **4**, 286 (Parent) 2) R.N. Jones & G.D. Thorn, CanadJRes **27B**, 831, 856(1949) & CA **44**, 2848f (1950)

N-Ethanolgluconamide or *N-(2-Hydroxyethanol)-gluconamide*,
$HOCH_2(CHOH)_4CONH.CH_2.CH_2OH$; mw 239.23, mp 109°, $[\alpha]_D^{20}$ +34.15°. It was prepd by refluxing ethanolamine with a gluconic lactone derived from date seed

Refs: 1) Beil, not found 2) K.J. Goldner & C.H. Rogers, JAmPharmAssoc **28**, 364–9(1939) & CA **33**, 7278(1939)

N-Ethanolgluconamide Hexanitrate,
$O_2N.O.CH_2(CHONO_2)_4CONH.CH_2.CH_2.ONO_2$; mw 509.21, N 16.50% ($NO_2$), OB to CO_2 −7.85%. It was prepd by mixed acid nitration of the preceding entry (Ref 2); no props given

Refs: 1) Beil, not found 2) W.F. Filbert, USP 2443903(1948) & CA **43**, 1796i(1949)

Ethanohydroxyacetamide and Derivatives

N-Ethanolhydroxyacetamide or *N-(2-Hydroxyethyl)-glycolamide*,
$HOCH_2CONH.CH_2.CH_2OH$; mw 119.11, mp 71–2°, bp 195–200°(1.5 torr, dec). It was prepd by heating ethanolamine with methyl hydroxyacetate

Refs: 1) Beil, not found 2) F.M. Meigs, USP 2347494(1944) & CA **39**, 1176(1945) 3) Q.F. Soper et al, JACS **70**, 2837–43 & CA **43**, 3364f (1949)

N-Ethanolhydroxyacetamide Dinitrate,
$O_2N.O.CH_2.CONH.CH_2.CH_2.ONO_2$; mw 209.12, N 13.39% ($NO_2$), OB to CO_2 −34.4%; prepd by mixed acid nitration of the preceding entry (Ref 2); no props given. It was proposed for use in blasting caps & detonators (Ref 2)

Refs: 1) Beil, not found 2) W.F. Filbert, USP 2443903(1948) & CA **43**, 1796i(1949)

Ethanolmethylethylenediamine and Derivatives

N-Ethanol-N-methyl-ethylenediamine or *1-Hydroxy-3,6-diazaheptane*,
$HO.CH_2.CH_2–NH.CH_2.CH_2.NH–CH_3$; mw 118.18. It can be made from N-methylethylenediamine by adding ethylene oxide (Ref 3)

N-Ethanolnitrate-N-methyl-ethylenedinitramine, called in Canad paper: **1-Nitroxy-3,6-dinitro-3,6-diazaheptane**,
$(O_2NO).CH_2.CH_2.N(NO_2).CH_2.CH_2.N(NO_2).CH_3$, mw 253.17, N 16.60% ($NO_2$), OB to CO_2 −53.7%, mp 90.5–92°(Ref 3); prepd indirectly from the parent by nitration followed by cleavage of a coupling compd (Ref 3)

Refs: 1) Beil – not found 2) R.N. Jones & G.D. Thorn, CanadJRes **27B**, 831 & 857(1949) & CA **44**, 2848(1950) (UV spectrum) 3) A.T. Blomquist & F.T. Fiedorek, USP 2481283 (1949) & CA **44**, 4926(1950)(Prepn)

Ethanolnitramine. See 1-Nitraminoethanol in Vol 1 of Encycl, p A200-R, under Aminoethanol

Ethanol Nitrilo and Derivatives

Ethanol Nitrilo or *Triethanolamine*,
$N(CH_2.CH_2.OH)_3$; mw 149.19, bp 206–207°(15 torr), 277–79°(150 torr), sp gr 1.1242, n_D^{20} 1.4852, sol in $CHCl_3$, hygroscopic. It was prepd by action of ammonia on 2-chloroethanol, or on ethylene oxide

Nitrilotrinitroethanol or **Tris(2-nitroxyethyl)-amine**, $N(CH_2.CH_2.ONO_2)_3$; mw 284.18, N 14.79% (NO_2), OB to CO_2 −50.7%. It was prepd by nitration of the alcohol, neutralization with $NaHCO_3$, extraction in ether (Ref 5). Thin layer chromatograph R_F values given in Ref 7

Nitrilotrinitroethanol nitrate or **Tris(2-nitroxyethyl)amine nitrate**, $HNO_3.N(CH_2.CH_2.ONO_2)_3$; mw 347.20, N 16.14% ($NO_2$), OB to CO_2 −30.0%, mp 74–75° dec (polymorph mp 64–5°, Ref 4), extremely unstable, dec violently; prepd by nitrating the alcohol in Ac_2O at −5 to −10°

Refs: 1) Beil **4**, 285, [729] (Parent) 2) Dynamit AG vorm Nobel & Co, BritP 350293 (1929) & CA **26**, 5423²(1932) 3) J. Barbière, BullSocChim **11**, 470–80(1944) & CA **40**, 2110 (1946) 4) G.E. Dunn, R.H. Meen & G.F. Wright, JACS **74**, 1344–45(1952) & CA **47**, 12394h(1953) 5) J. Metadier, FrP 984523

(1951) & CA **49**, 11005i(1955) 6)L. Médard, MP **36**, 93–6(1954) & CA **50**, 6795(1956) 7) R.W. Barnes, JChromatogr **31**(2), 606–8 (1967) & CA **68**, 117167(1968)

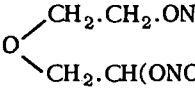
Nitrilotrinitroethanol Phosphate, $N(CH_2.CH_2.ONO_2)_3.2H_3PO_4$; mw 498.32, N 16.24%, OB to CO_2 −28.9%, mp 108–10°(dec), sol in alc. It was prepd by adding ethereal H_3PO_4 to ethereal Nitrilotrinitroethanol (Refs 1 & 2)
Refs: 1) Beil – not found 2) J. Metadier, FrP 984523(1951) & CA **49**, 11005i(1955)(Prepn) 3) K. Junkmann, H.H. Inhoffen, S. Grünler & E. Neuhoff, GerP 830955(1952) & CA **52**, 11109i (1958)(Prepn) 4) M.D. Mashkovskii & B.A. Medvedev,MedProm(Russ)**12**, No 4, 56–7(1958) & CA **53**, 20501g(1959)(Props)

Ethanol-2,2′,2″-nitrilotrinitrate, Dimer or **1,1,4,4-Tetrakis(2-hydroxyethyl)piperazinium Dinitrate, Tetranitrate,** $C_{12}H_{24}N_8O_{18}$; mw 568.37, N 14.79%(NO_2), OB to CO_2 −50.7%, mp 216°(dec). It was prepd from nitrilotrinitroethanol nitrate and base at 0°.
Refs: 1) Beil – not found 2) G.E. Dunn, R.H. Meen & G.F. Wright, JACS **74**, 1344–45 (1952) & CA **47**, 12394i(1953)

Ethanol, Nitroxytrinitro. See Ethanol-2;2,2-trinitro-1-nitrate under Ethanol Derivatives

Ethanoloxypropanediol and Derivatives
Ethanol-2-oxypropane-2,3-diol, $HO.CH_2.CH_2.O.CH_2.CH(OH).CH_2.OH$; mw 136.15, bp 145–50°(1 torr), 162–4° (3 torr), n_D^{20} 1.4723, sp gr 1.218, heat of combustion 4946.5cal/g. It was prepd by reacting ethylene glycol with epichlorohydrin and hydrolyzing the intermediate (Ref 2)
Ethanol-2-oxypropane-2,3-diol Dinitrate, $HO.CH_2.CH_2.O.CH_2.CH(ONO_2)CH_2.ONO_2$; mw 226.14, N 12.39%, OB to CO_2 −49.6%. It was probably prepd by partial nitration of the parent; no mention in the open literature
Refs: 1) Beil – not found 2) M.S. Kharasch & W. Nudenberg, JOC **8**, 189–93(1943) & CA **37**, 3736(1943) 3) L. Médard & M. Thomas, MP **37**, 129(1955) & CA **51**, 716h(1957)

Ethanol-2-oxypropane-2,3-diol Trinitrate,



$CH_2.CH(ONO_2).CH_2(ONO_2)$; mw 271.14, N 15.50%, OB to CO_2 −26.5%, sp gr 1.481, n_D^{20} 1.4685, heat of combustion 2405.1cal/g. It was prepd by nitration of parent with mixed acid at room temp (Ref 4)
Refs: 1) Beil – not found 2) L. Médard & M. Thomas, MP **37**, 129–30 & 138(1955) & CA **51**, 716(1957) 3) G. Desseigne, MP **39**, 181–90(1957) & CA **52**, 21107e(1958)(Prepn) 4) G. Desseigne, FrP 1127647(1956) & CA **53**, 16970d(1959)

Ethanol Perchlorate, $HO.CH_2.CH_2.OClO_3$; mw 144.51, OB to CO_2 −16.6%, sp gr more than 1.7, sol in Et_2O, sl sol in w, dec violently on heating, impact, grinding; prepd by adding ethylene glycol to perchloric acid at −75 to −78°(Ref 3). Power by Trauzl Test 110% PA or 120% TNT. It is extremely sensitive
Refs: 1) Beil – not found 2) ADL, PureExpl-Compds, Part 1(1947), p 62 3) A.A. Zinov'ev, I.A. Zakharova & G.P. Kondratskaya, ZhurNeorg-Khim **3**, 2390–4(1958) & CA **55**, 208f(1961)

Ethanol Tetranitroanilino Nitrate or **Pentryl.** See 2-(2′,4′,6′-Trinitro-N-nitranilino)-ethanol Nitrate or 2-(N,2,4,6-Tetranitroanilino)-ethanol Nitrate in Vol 1 of Encycl, pp A425 to A429

Ethanthiol. See Ethylmercaptan

Ethene. See Ethylene

Ethenoxyethene. See Divinyl Ether or Divinyl oxide in Vol 5 of Encycl, p D1526-L

Ethenylamidine or **Ethanamidine.** See α-Amino-α-imidoethane in Vol 1 of Encycl, p A223-L

Ethenylamidoöxime and Derivatives
Ethenylamidoöxime or *Acetamidoxime,* $CH_3.C(=NOH)NH_2$; mw 74.08, N 18.77%(NO),

mp 135°(dec), sol in w and alc, dec in warm w. Prepd by addn of H_2NOH to CH_3CN
Refs: 1) Beil **2**, 188 2) J. Barrans, R. Mathis-Noel & F. Mathis, CR **245**, 419–22 (1957) & CA **52**, 882(1958)(IR spectrum)

Nitroethenylamidoöxime, $O_2N.CH_2.C(=NOH)NH_2$; mw 119.08, N 23.52% (NO), OB to CO_2 −47.0%, yel cryst, dec 108°, sl sol in hot alc; prepd from the ammonium salt of nitroacetonitrile and hydroxylamine hydrochloride (Ref 2)
Refs: 1) Beil **2**, 227 & (100) 2) W. Steinkopf & L. Bohrmann, Ber **41**, 1050(1908)

Some unstable derivs of Ethenylamidoöxime include:

N-Hydroxyethenylamidoöxime, $CH_3C(=NOH)NH.OH$; mw 90.08, N 31.10%, oil, dec explosively when dry. Prepd by sodium amalgam reduction of ethyl nitrolic acid (Ref 1)
Refs: 1) Beil **2**, 189 2) J. Armand, CR **258**(1), 207–10(1964) & CA **60**, 10498(1964) 3) Ibid, **262**(7), 592–5(1966) & CA **64**, 19406(1966) (Prepn of the HCl salt) 4) F. Valentini, P. Gouzerh & P. Souchay, JChimPhysPhysicochimBiol **1971**, 68(4), 601–4 & CA **75**, 54125m (1971)

N-Oxoethenylamidoöxime (Äthylnitrosolsaüre in Ger), $CH_3C(=NOH)NO$; mw 88.06, N 31.81%, OB to CO_2 −72.7%, unstable in free state; prepd from following entry by reduction with sodium amalgam
Refs: 1) Beil **2**, 189 2) H. Wieland, Ann **353**, 90(1907) 3) J. Armand, CR **258**(1), 207–10 (1964) & CA **60**, 10498(1964)(Prepn) 4) F. Valentini, P. Gouzerh & P. Souchay, JChimPhysPhysicochimBiol **1971**, 68(4), 601–4 & CA **75**, 54125m(1971)

N-Dioxoethenylamidoöxime, (Äthylnitrolsäure in Ger), $CH_3C(=NOH)NO_2$; mw 104.065, N 26.92%, OB to CO_2 −46.1%, rh cryst, mp 81–2°(dec), 87–8°, very sol. It was prepd from nitric acid and sodium salt of nitroethane (Ref 2)
Refs: 1) Beil **2**, 189 2) V. Meyer, Ann **175**, 98(1875) 3) A.I. Ivanov et al, ZhOrgKhim **2**(5), 763–6(1966) & CA **65**, 12098(1966) (UV, IR spectra)

O-1-(1,1-dinitroethane)ethenylamidoöxime, $CH_3C[=NO.C(NO_2)_2CH_3]NH_2$; mw 192.13, N 21.87%(NO), OB to CO_2 −58.3%, mp 95–97° dec, wh ndls, not stable prepn is in Ref 2
Refs: 1) Beil – not found 2) J.S. Belew, C.E. Grabiel & L.B. Clapp, JACS **77**, 1110–14 (1955) & CA **50**, 1648(1956)(Prepn) 3) A.I. Ivanov et al, ZhOrgKhim **2**(5), 763–7(1966) & CA **65**, 12098(1966) (UV, IR spectra)

N-Dioxo-O-1-(1,1-dinitroethane)ethenylamidoöxime or **2,5,5-Trinitro-3-aza-4-oxa-2-hexene,** $CH_3C[=N-O-C(NO_2)_2CH_3]NO_2$; mw 222.115, N 25.22%, OB to CO_2 −28.8%, mp 121.2–121.6°, wh pltlts. It was prepd by reacting NH_4 or K salt of 1-nitroethanenitronate with 1,1-dinitroethane (use at most 5g of salt) (Ref 1); inert to hot nitric acid; does not form a perchlorate salt
Refs: 1) J.S. Belew, C.E. Grabiel & L.B. Clapp, JACS **77**, 1110–14(1955) & CA **50**, 1648(1956) (IR spectrum) 2) A.I. Ivanov et al, ZhOrgKhim **2**(5), 763–6(1966) & CA **65**, 12098(1966)(UV & IR spectra)

Ethenyldiphenylamidine. See N,N′-Diphenylacetamidine in Vol 5 of Encycl, p D1414-R

Ether, Diethyl; Ethyl Ether or **Sulfuric Ether.** See Diethyl Ether in Vol 5 of Encycl, pp D1233 to D1235, where its preparation, properties and some qualitative tests are described. Its requirements and tests, as outlined in US Specification MIL-E-199A(1), are not described there, but are described below
Uses of Ether. One of the important applications of ether is in manuf of single-base propellants where it is used in 2:1 mixture (by wt) with ethanol. The procedure is described in Vol 3 of Encycl, p C399, under Colloiding Agents and Colloidal Propellants

Another important use of ether is in analyses for extraction of materials soluble in it. For example, in extraction of nitrocompounds, known as "Nitrobodies" (NB), always present in spent acids of TNT manuf, the following procedure is described in Clift & Fedoroff's Manual for Explosives Laboratories, Lefax, Inc, Phila, Pa, Vol **1**(1943), Chap 1, Pt 3, p 21–2: Shake the bottle contg the spent acid and pour 50.00g into a tared porcelain or Pyrex dish of about 100ml capacity, using a pulp balance. If the acid solidified due to an excessive amt of NB's,

warm the bottle in water prior to pouring. Transfer the sample from the dish into a 400ml beaker con tg 200ml ice-water mixture and then into a 500ml separatory funnel. Add 50ml of ether and remove the funnel from the support, while holding the stopcock firmly with the right hand. Close the top of the funnel with a ground glass stopper and, while pressing against it with the palm of the left hand, carefully invert the funnel. Immediately after this, open the stopcock to allow the vapors of ether to escape. Close the stopcock and, while holding the funnel in an inverted position, shake the mixture gently and then open the stopcock, being careful that none of the liquid escapes. Repeat the shaking operation 2 or 3 more times and then replace the funnel on the support. After allowing the funnel to stand until the two layers separate, open the stopcock and draw off the bottom layer (acid-water) into the same 400ml beaker as above. Allow a few drops of the ether to enter the beaker to avoid leaving any acid in the ether layer and then close the stopcock. Place the tared dish used for weighing the 50g sample of acid, under the stopcock of funnel and draw off the ether layer into the dish. Place the dish on a water bath and, while the ether evaporates, pour the acid-water from 400ml beaker into the same separatory funnel. Add 50ml of ether and extract NB's in the same manner as above and transfer the ether extract into the above dish after nearly all the ether of the 1st extraction has been evaporated. Repeat the extraction and separation using 25ml of ether, etc. The extracted material in the dish should be heated until complete disappearance of ether odor, then cool the dish in a desiccator and weigh

$$\% \text{ NB} = \frac{\text{Wt of residue in the dish x 100}}{\text{Wt of sample}}$$

Note: As small amounts of acid may be retained in the ether extracts, it might be advisable (for more precise analysis) to collect the three ether extracts in a 250ml separatory funnel, wash with about 50ml water, and then proceed with the evapn of ether

Ether, Diethyl. US Armed Forces Specification Requirements and Tests for material used in manuf of smokeless proplnts and for cleaning optical instruments is covered by MIL-E-199A(1) with AMD 2(MU), 3 June 1968. Their contents are as follows:

1. *SCOPE*

1.1. This specification covers one grade of ether

2. *APPLICABLE DOCUMENTS*

2.1. A list of documents which includes Federal and Military Specifications, Ordnance Corps Publications, etc is given in the above Spec

2.2. Other publications, which include "Interstate Commerce Commission Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles", and ASTM (American Society for Testing and Materials) **D1209-69** publication, entitled "Standard Method of Test for Color of Clear Liquids" (Platinum-Cobalt Scale) are listed in the same Spec

3. REQUIREMENTS

3.1. **Material.** Diethyl ether shall be made from ethyl alcohol conforming to the requirements for grade 1 or grade 2 of Specification MIL-A-463 (See in this Vol under Ethanol), or ether shall be made as a co-product in the manuf of Ethanol from ethylene

3.2. **Appearance.** Ether shall be no darker than the 20 Platinum-Cobalt Standard when tested as specified in paragraph 4.3.1

3.3. **Chemical and Physical Properties.** The diethyl ether shall conform to the requirements specified in Table Et 3

Table Et 3

Chemical and Physical Requirements

Specific gravity at 20/20°C	0.712 to 0.723
Nonvolatile residue, % by weight, maximum	0.002
Acidity, as acetic acid, %, max	0.006
Acetylene, %, max	0.001
Peroxides	None
Chlorides	None
Aldehydes	None
(Not applicable when ether is intended for use in manuf of smokeless propellants)	

4. QUALITY ASSURANCE PROVISIONS are described on pp 2–7

This includes among other items the description of sampling from 55-gallon metal containers or from tank cars in which ether is delivered to Ordnance Installations *4.2.3.1.1. Containers* (p 5). Select from each inspection lot two containers at random and

remove from each about 16 oz, to a bottle provided with a glass stopper, thus making a 32-oz composite sample. Label the bottle

Note: A lot shall consist of one or more batches of ether, produced by one manufacturer, under one continuous set of operating conditions and submitted for acceptance at one time. Each batch shall consist of that quantity of ether that has been subjected to the same unit chemical or physical mixing process intended to make the final product homogeneous

4.2.3.1.2. Tank Cars. Attach a clean, tared, small-necked glass bottle of about one quart size to a rod equal in length to the diam of tank car. Lower the unstoppered bottle quickly inside the tank to its bottom and start immediately to withdraw it to the surface. In order to obtain a representative cross-section of the material, the speed of lowering and raising the bottle shall be uniform and so regulated that the bottle is just filled as it reaches the surface of ether in the tank. Transfer the sample to a glass bottle provided with a glass stopper and attach a label

4.3. Test Method and Procedure

4.3.1. Appearance. It shall be detd in accordance with ASTM Designation **D1209–69**, entitled "Standard Method of Test for Color of Clear Liquids" (Platinum-Cobalt Scale)

Apparatus:

a) Beckman Model B Spectrophotometer or any other instrument having equivalent resolution and sensitivity

b) Spectrophotometer Cells, matched, having a 10-mm light path

c) Color Comparison Tubes. Matched 100-ml tall-form Nessler tubes, provided with ground-on optically clear glass caps. The height of the 100-ml mark should be 275 to 295mm above the bottom of the tube

d) Color Comparator. It shall be constructed to permit visual comparison of light transmitted thru the above Nessler tubes in the direction of their longitudinal axes. The white light shall pass thru or be reflected off a white glass plate and directed with equal intensity thru the tubes and shall be shielded so that no light enters the tubes from the side

Reagents:

a) Water must be "reagent grade" conforming to ASTM Spec D1193

b) Cobalt Chloride, $CoCl_2.6H_2O$, Hydrochloric Acid (sp gr 1.19) and Potassium Chloroplatinate, K_2PtCl_6, shall conform to ASTM Method E200

c) Platinum-Cobalt Stock Solution. Dissolve in 1-liter volumetric flask 1.245g of K_2PtCl_6 and 1.000g of $CoCl_2.6H_2O$ in about 800ml of reagent-grade water, add 100ml HCl, and dilute to 1 liter with water. The absorbance of this "stock solution" is supposed to be 500 but it should fall within the limits given in Table Et 4 for various wavelengths. The measurements shall be made in a cell having a 10mm light path using a Beckman Model B Spectrophotometer at a sensitivity setting of one, or in an equivalent spectrophotometer. Reagent water in a matched cell shall be used as the reference soln

Table Et 4

Wavelength, nm	Absorbance
430	0.110 to 0.120
455	0.130 to 0.145
480	0.105 to 0.120
510	0.055 to 0.065

Platinum-Cobalt Standards. By diluting 1, 2, 3, 4, 5 and 6ml, each to 100ml with reagent water, Color Standards Nos 5, 10, 15, 20, 25 and 30, respectively, are obtd. Table 2 of D1209-69 Standards lists several more concentrated standards up to Std No 500, but for Ether of US Spec they are not required, since its color shall not be darker than No 20 Standard

Procedure:

Introduce 100ml of ether under test into a Nessler tube, passing the sample thru a filter if it has any visible turbidity. Cap the tube with a transparent cover and place it in the "comparator", while side-by-side with it is placed one of the standards. Report as the color the number of the standard that most nearly matches the sample. In the event that the color lies midway between two standards, report the darker of the two. If, owing to differences in hue betw the sample and the standards, a definite match cannot be obtd, report the range over which an apparent match is obtd, making a note that it is "off-hue"

4.3.2. Specific Gravity

4.3.2.1. Pycnometer Method. Sp gr shall be detd in accordance with Federal Test Method No 141 Method 4183, except that the temperature shall be 20/20°C

Note: Pycnometer Method is also described

in Vol 3 of Encycl, pp D69 to D71

4.3.2.2. Westphal Balance Method (Alternative Method). Sp gr shall be detd in accordance with Federal Test Method No 141 Method 4183, except that the temp shall be 20/20°C

Note: Westphal Balance Method is described under Hydrostatic Weighing Method in Vol 3 of Encycl, p D68

4.3.3. Nonvolatile Residue. Transfer 100ml of the sample to a tared dish and after evaporating to dryness on a steam bath, place the dish for one hour in an oven at 100°. Cool in a desiccator, weigh and calculate

$$\% \text{ Nonvolatiles} = 100A/BC,$$

where:

A = wt of residue in grams
B = volume of sample and
C = sp gr of sample

4.3.4. Acidity. Prepare the bromothymol-blue indicator soln by dissolving in a 100ml volumetric flask 100mg of bromothymol blue in 20ml of acid-free alcohol and adding distd water to make 100ml of soln. Introduce into a colorless flask provided with a tightly-fitting glass stopper, 2 drops of the above indicator soln and add dropwise from a 25ml burette 0.001N NaOH soln to the first blue color that remains permanently developed. Add 50ml of the sample, shake well to mix the two layers and titrate with 0.001N NaOH soln to the first blue color that persists for several mins. Close the bottle (to prevent introducing any carbon dioxide from the atm); wait until two layers separate and then judge the color. Small amts of alcohol in the soln dim the color, while large amts give an indefinite end point. Opalescence occurs when ether is slightly alkaline. Calculate acidity as % of Acetic Acid

$$\% \text{ Acetic Acid} = (6.0VN)/AB,$$

where:

V = ml of NaOH soln used in titration
N = normality of NaOH soln
A = ml of sample used
B = sp gr of sample

4.3.5. Peroxides. Transfer 10ml of the sample to a 10ml colorless cylinder, provided with a glass stopper, add 1ml of soln prepd by dissolving 0.5g KI and 0.5g CdI_2 in 9ml of distd water. Stopper the cylinder and keep it in a dark place for 1 hour, while shaking occasionally. If ether layer darkens, peroxides are present

4.3.6. Chlorides. Transfer 10ml of the sample to a test tube and add 4 to 5ml of a 10% silver nitrate soln. If an opalescence appears, introduce one drop of nitric acid by means of a stirring rod. Persistence of opalescence indicates the presence of chlorides

4.3.7. Aldehydes. Prep the reagent by mixing 1ml of alkaline mercuric potassium iodide with 17ml of a satd aq soln of NaCl. Transfer 10ml of the sample to a colorless cylinder, provided with a glass stopper and add 1ml of the above reagent. Stopper the cylinder, shake vigorously for 10 secs and then set it aside for 1 min. Persistence of turbidity in the aqueous layer indicates the presence of aldehydes

Note: This test is not required for ether intended for use in the manuf of smokeless proplnts

4.3.8. Acetylene. Add 10 drops of freshly prepd 0.1% methyl red indicator soln to each of two 250ml Erlen flasks contg 50ml of 50% ethanol soln. Reserve one of the flasks as a blank and add 10ml of the sample to the other. Neutralize the acidity in each of the flasks and match their colors exactly by adding to each flask 0.02N alcoholic silver nitrate soln and then titrate the sample and blank with standard 0.02N NaOH soln to the same yellow end point. Calculate as follows:

$$\% \text{ Acetylene} = 1.30(V-v)N/SD$$

where:

V = ml std NaOH soln required for the sample
v = ml std NaOH soln required for the blank
N = normality of NaOH soln
S = ml of sample
D = sp gr of sample

Determination of Ether in Mixtures with Ethanol, Acetone and Water

Following are the titles of procedures described or listed under ETHANOL

1) Ethanol, Ether, Acetone and Water Determinations in Solvents Used in Manufacture of Smokeless Propellants, as Described in the Literature

2) Ethanol, Ether, Acetone and Water Determinations in Nitrocelluloses and Smokeless Propellants as Described in the Literature

3) Ethanol + Ether as "Residual Solvent" Determination in Single-Base Propellants

4) Ethanol, Ether and Water Determination in Single-Base Propellants by US Army Munitions Command Method T103.5, entitled "Total Volatiles, Gas Chromatographic Method"

Following are additional references on analytical procedures involving ether:
A) Anon, IEC, NewEdn, **14**, 305(1936)(Peroxide formation in ethers and a test for peroxides in ether) [*Procedure:* a) Add 10ml ether (sample to test) to 150ml of 2N sulfuric acid soln, followed by 3 drops of 1% soln of Amm molybdate (a catalyst to favor the liberation of dine) and 15ml of 10% KI soln. Shake well and allow to stand for 15 mins b) Titrate the liberated iodine with 0.05N Na thiosulfate and shake well after each addn until near discoloration of soln c) Add a few cc of starch soln and continue titration until disappearance of blue color]
B) A. Boulègue, MémServChimÉtat, **36**, F2, p 258(1951) (Determination of ether by oxidation with K dichromate. Detn of the carbon dioxide formed by combustion of such hydrocarbons as ether & acetone was shown to be a suitable method for estimating 0.5–10mg quantities in the atm
C) H. Liogier, MP **38**, 167–72(1956) & CA **51**, 11719(1957)(Determination of ether in waste alcohols. A 5–8g sample is suspended in 200ml $HOCH_2.CH_2OH$ contg 1% Ph_2NH. Under a vacuum of 40–50cm of water created by a Mariotte bottle, the mixt is heated at 95° with stirring until the sample dissolves or degelatinizes. While heating 850ml of water is added slowly to ppt nitro compds. About 250ml of water is distilled into an intermediate flask under cooling. This flask is warmed almost to boiling and 1½ liters of air passed thru over 1½ hrs, entraining the ether which is passed thru a reflux condenser and collected in 30ml 1.6N H_2CrO_4 contd in a bubble tube in series with the Mariotte bottle. The stillpot residue is distd and the alc collected in another bubbler contg H_2CrO_4 soln. The bubbler contents are titrated with thiosulfate soln and ether & alc calculated)
D) Standard Methods of Chemical Analysis, 6th Ed **2**, 483–88, F.J. Welcher, Ed (1963)

Éther à 56°. Fr designation of ether-ethanol mixture of 56 degrees Baumé. It consists of ether 64 & ethanol 36% (by weight), which makes about 2 parts of ether and 1 part of ethanol, by volume. Such a mixt has been used for gelatinization of NC of ca 12.6% N during manuf of single-base proplnts not only in France, but also in the USA and other countries

Éther amylazotique or **Éther amylnitrique.** Fr for Amyl Nitrate described in Vol 1, p A397-L

Éther éthylacétique. Fr for Ethyl Acetate, described in this Vol

Éther éthylazotique or **Éther éthylnitrique.** Fr for Ethyl Nitrate

Éther étylique. Fr for Ethyl Ether, described in Vol 4 as Diethyl Ether, p D1233

Éther méthylazotique or **Éther méthylnitrique** Fr for Methyl Nitrate

Ether-oxyline Explosives, patented in England in 1852 & 1853 by Winiwarter, were based on "ether-oxyline" which was a soln of Pyroxylin in 2 parts by wt of ether. It was presumably used as a binder. Daniel (Ref 1) lists the following compositions mixed with "ether-oxyline":
No 1: MF, K chlorate, Sb sulfide, charcoal, saltpeter, K ferrocyanide & Mn dioxide
No 2: Zn fulminate, K chlorate, Sb sulfide, Pb dioxide and K ferrocyanide
No 3: Phosphorus (amorphous), Pb dioxide, charcoal & saltpeter

Exact compns were given in Ref 2
Refs: 1) J.P. Cundill, MP **6**, 118(1893)(Gives details of compns) 2) Daniel, Dict(1902), 810(Under Winiwarter)

Ethoxyacetamide (Äthoxyacetamid or Äthylätherglykolsäureamid, in Ger), $C_2H_5O.CH_2CONH_2$; mw 103.12, N 13.58%; plates (from benz); mp 82.5°(sublimes), bp 225° at 75mm Hg; easily sol in w, alc, eth, benz and CS_2. It can be prepd by action of aqueous ammonia on ethyl ester of ethoxyacetic acid or by action of ammonia on acetoxyacetyl chloride in ether

Its uses are not indicated
Refs: 1) Beil **3**, 241, (93) & [174] 2) W. Heintz, Ann **129**, 39(1864) 3) T.L. Brown, J.F. Regan, R.D. Schuetz & J.C. Sternberg, JPhysChem **63**, 1324–5(1959) & CA **54**, 1073 (1960)(Spectrum)

Ethoxyacetazide or **Ethoxyacetic Acid Azide,** $C_2H_5O.CH_2.CO(N_3)$; mw 129.12, N 32.54%, yel pungent oil, causing tears; sol in alc, eth, benz; expl on heating; prepd from ethoxyacetic acid hydrazide and HNO_2
Refs: 1) Beil **3**, (94) 2) T. Curtius, JPraktChem [2] **95**, 174(1917) & CA **12**, 899(1918)

Ethoxyacetanilide. See Acetamidophenetole in Vol 1 of Encycl, p A20

Ethoxyacetylene or **Ethyl ethynyl ether,** $C_2H_5O.C{:}CH$; mw 70.09, bp 50–52°, n_D^{20} 1.3796, sp gr 0.799, dipole moment 1.94D, irritating to eyes. It hydrates explosively with concd HCl, controllably with dil HCl; unstable at room temp; reacts with CCl_4 slowly

Its **sodium** derivative, presumably $C_2H_5O.C{:}CNa$, explodes when exposed to air. It can be prepd from sodamide in liquid ammonia and diethylchloracetal
Refs: 1) Beil – not found 2) T.L. Jacobs, R. Cramer & J.E. Hanson, JACS **64**, 223–6 (1942) & CA **36**, 1899(1942)(Heated bromoethoxyethylene with KOH at 90–100°; bp 28° at 300mm, n_D^{20} 1.3812, sp gr 0.793) 3) A.E. Favorskii & M.N. Shchukina, JGenChem **15**, 394–400(1945) & CA **40**, 4657(1946) (Mentions Ag salt, wh amor, explosive) 4) D.A. van Dorp, J.F. Arens & O. Stephenson, RecTravChim **70**, 289–96(1951) & CA **45**, 7950(1951) (Heated Et2-chlorovinyl ether with KOH at 120°, bp 50–52°) 5) J. Queck, AbhandlBraunschweigWissGes **4**, 37–47(1952) & CA **48**, 1238(1954)(Review on prepn, reactions) 6) T.R. Rix & J.F. Arens, KoninklNedAkadWetenschap, Proc **56B**, 364–7(1953) & CA **49**, 2300(1955)(Reacts with picric acid to give Et picryl ether and picryl acetate) 7) E.R.H. Jones et al, OrgSyn **34**, 46–9(1954), JChemSoc **1954**, 1860–5 & CA **49**, 5265, 8142 (1955)(Prepn from $ClCH_2CH(OEt)_2$ and sodamide; mentions expl nature of the Na intermediate in contact with air) 8) Y.A. Sinnema & J.F. Arens, RecTravChim **74**, 901–4(1955) & CA **50**, 4925(1956)(Prepn of $CH_3C(N_3)_2OEt$ from EtOC≡CH and HN_3; mw 156.15, N 53.82%, bp 46–46.5°(10 torr), n_D^{20} 1.4464) 9) J. Cley & J.F. Arens, RecTravChim **78**, 929–34(1959) & CA **54**, 18349(1960)(Prepn of an unstable, expl material with propiolic acid) 10) L. Weinberger, ChemProd **26**(11), 23–7(1963) & CA **60**, 3994(1964) (Review, mentions explosivity of dry Na and Li salts in air)

Ethoxyaminobenzenes. See Aminophenetols in Vol 1 of Encycl, p A240

2-Ethoxy-2-nitramino-1-nitroimidazolidine, $C_5H_{11}N_5O_5$; mw 221.17, N 12.66%(NO_2), OB to CO_2 –76.0%, mp 133.6–134°; prepd by refluxing 1-nitro-2-nitraminoimidazolidine in EtOH (Ref 2)
Refs: 1) Beil – not found 2) A.F. McKay & G.F. Wright, JACS **70**, 3990–4(1948) & CA **43**, 2203(1949)

Ethoxyanilines. See Aminophenetols in Vol 1 of Encycl, p A240

Ethoxybenzazide or **p-Ethoxybenzoyl Azide,** $C_2H_5O.C_6H_4CO(N_3)$; mw 191.19, N 21.98%, solid, mp 31° and explodes at higher temp. It was prepd by Curtius & Ulmer (Refs 1 & 2) by boiling the Et ester with hydrazine, then treatment with HNO_2
Refs: 1) Beil, not found 2) Th. Curtius & W. Ulmer, JPraktChem **125**, 58(1930) & CA **24**, 3232(1930) 3) P.P.T. Sah & K-S Chang, Ber **69B**, 2762–4(1936) & CA **31**, 2180(1937) (Prepn from hydrazide)

Ethoxydiphenylamine and Derivatives
Ethoxydiphenylamine (ortho), $C_6H_5NH.C_6H_4(OC_2H_5)$; mw 213.28, bp 135–6° (½–1 torr) and 142–6°(3.5 torr), sp gr 1.095, n_D^{20} 1.6202. It is prepd by heating aniline, resorcinol and calcium chloride (Refs 2 & 3)

and by reducing the vinyl ether resulting from adding acetylene to the phenol (Ref 4)
Refs: 1) Beil – not found 2) A. Calm, Ber **16**, 2786(1883) 3) K. Palat et al, Československ-Farm **8**, 569–71 & CA **54**, 22424(1960) 4) B.I. Mikhant'ev & L.V. Radzhyunas, Isv-VysshikhUchebnZavedenii, KhimIKhimTekhnol **6**(4), 697–8(1963) & CA **60**, 2810(1964)

Ethoxydiphenylamine (meta), mp 59–60°, bp 154–61°(2 torr) & 170.5–172°(2.5 torr). See ortho for prepn
Refs: 1) Beil **13**, 411 2–4) See under ortho

Ethoxydiphenylamine (para), ndls, mp 71–2° and 73–4°, bp 348° and 145°(0.9 torr), sol in eth, benz; prepd by refluxing p-hydroxy-diphenylamine with C_2H_5I and $NaOC_2H_5$ (Ref 2)
Refs: 1) Beil **13**, 446 2) P. Jacobson et al, Ber **26**, 696(1893) 3) L.G. Makarova, IzvestAkadNaukSSSR, OtdelKhimNauk **1951**, 741–4 & CA **46**, 7532(1952)(Prepn) 4) K. Palat, A. Sekera & C. Vbrz, ChemListy **51**, 563–78 & CA **51**, 10404(1957)(Prepn) 5) B.I. Mikhant'ev & L. Radziunas, ZhObshch-Khim **34**(10), 3424–5(1964) & CA **62**, 2727 (1965)(Prepn) 6) Monsanto Co, BritP 975097 & CA **62**, 5227(1965)(Prepn)

Ethoxydiphenylamine (ortho), o,p-Dinitro, $C_6H_5.NH.C_6H_2(NO_2)_2.OC_2H_5$; mw 303.28, yel ndls, mp 155°; prepd from aniline and 2,3,5-trinitrophenetole
Refs: 1) Beil **13**, 393 2) J.J. Blanksma, RecTravChim **24**, 41(1905)

Ethoxydiphenylamine (ortho), o',p'-Dinitro, $(O_2N)_2C_6H_3.NH.C_6H_4.OC_2H_5$; mw 303.28, red ndls (from alc), mp 164° and 172–3°; prepd by refluxing C_2H_5I with the Na derivative of the corresponding phenol (Ref 2)
Refs: 1) Beil **13**, 366 2) M. Schöpff, Ber **22**, 902(1889) 3) H.J.vOpstall, RecTravChim **52**, 901–11(1933) & CA **28**, 26(1934)

Ethoxydiphenylamine (meta), o,o-Dinitro, $C_6H_5.NH.C_6H_2(NO_2)_2.OC_2H_5$; mw 303.28, yel cryst, mp 125°. It is prepd from aniline and 2,3,4-trinitrophenetole
Refs: 1) Beil **13**, 423 2) J.J. Blanksma, RecTravChim **27**, 54(1908)

Ethoxydiphenylamine (meta), o,p-Dinitro, formula same as above, mw 303.28, yel cryst, mp 170°. It was prepd from aniline and 5-chloro-2,4-dinitro-phenetole
Refs: 1) Beil **13**, 424 2) J.J. Blanksma, RecTravChim **23**, 123(1904)

Ethoxydiphenylamine (meta), o',p'-Dinitro, $(O_2N)_2C_6H_3.NH.C_6H_4.OC_2H_5$; mw 303.28, or-yel cryst, mp 151°, sol in acet, benz, hot alc. Prepn not given in Ref 2
Refs: 1) Beil **13**, (131) 2) Reverdin & Lokietek, Bl[4] **17**, 408 (from Beil)

Ethoxydiphenylamine (para), o',p'-Dinitro, formula same as above, mw 303.28, red pltlts (from alc), mp 119–20°. It was prepd from 2,4-dinitro-chlorobenzene and p-phenetidine
Refs: 1) Beil **13**, [232] 2) A. vBlom, Helv **4**, 1037(1921) & CA **16**, 901(1922)

Ethoxydiphenylamine (ortho), o',o',p'-Trinitro, $(O_2N)_3C_6H_2.NH.C_6H_4.OC_2H_5$; mw 349.085, N 12.04%($NO_2$), red ndls, mp 124–5° and 136–7°(perhaps configurational isomerism due to restricted rotation around the –NH–). It was prepd from o-phenetidine and picryl chloride (Ref 2) or picryl azide (Ref 3)
Refs: 1) Beil **13**, 366 2) M. Busch & E. Pungs, JPraktChem [2] **79**, 548, 533(1909) & CA **4**, 900(1910) 3) R. Andriseno & D.D. Casoni, BollSciFacoltaChimIndBologna **1943**, No 1, 1, 7 & CA **41**, 723(1947)

Ethoxydiphenylamine (meta), o,o,p-Trinitro, $C_6H_5.NH.C_6H(NO_2)_3.OC_2H_5$; mw 349.085, N 12.04%($NO_2$), mp 174°. It was prepd from aniline and 3-chloro-2,4,6-trinitro-phenetole
Refs: 1) Beil **13**, 425 2) J.J. Blanksma, RecTravChim **21**, 326(1902)

Ethoxydiphenylamine (para), o',o',p'-Trinitro, $(O_2N)_3C_6H_2.NH.C_6H_4.OC_2H_5$; mw 349.085, N 12.04%($NO_2$), red ndls, mp 123–4°, 136–7°, 138.5°, sol in alc & benz. It was prepd from p-phenetidine and picryl chloride (Refs 2 & 3) or Picryl Azide (Ref 4)
Refs: 1) Beil **13**, 446 2) See (2) under ortho-trinitro above 3) N.M. Cullinane, O.E. Embrey & D.R. Davies, JChemSoc **1932**, 2363–4 & CA **26**, 5926(1932) 4) See (3) under ortho-trinitro above

Ethoxyethanol and Derivatives

2-Ethoxyethanol, $C_2H_5O.CH_2.CH_2.OH$; mw 90.12, bp 134° (721.5 torr) and 134.7–139° (748.3 torr), sp gr ^{15}0.935, n_D^{20} 1.4080. It was prepd by warming C_2H_5I with Na in glycol; mentioned as solvent for cellulose nitrate in Ref 1, [518]

Refs: 1) Beil **1**, 467, (244) & [518] 2) E. Demole, Ber **9**, 745(1876) 3) M.H. Palomaa, Ber **42**, 3876(1909)

2-Ethoxyethanol Azide or *2-Azidoethyl Ethyl Ether*, $C_2H_5O.CH_2.CH_2.N_3$; mw 115.14, N 36.41%, colorless liq, bp 49°(25 torr), sp gr ^{24} 0.9744. It was prepd by action of Ag_2O on C_2H_5I and $N_3.CH_2.CH_2.OH$ (Ref 2). It is an expl compd

Refs: 1) Beil **1**, (171) 2) M.O. Forster & S.H. Newman, JChemSoc **97**, 2579(1910) 3) K.A. Korner & S.B. Serebryanii, Zapiski-InstKhim, AkadNaukUSSR, InstKhim **6**, Nos 3–4, 343–50(1940) & CA **35**, 2469(1941)

2-Ethoxyethanol Nitrate or *2-Nitratoethyl Ethyl Ether*, $C_2H_5O.CH_2.CH_2.ONO_2$; mw 135.12, N 10.37%, bp 64° (12 torr), sp gr ^{20} 1.1118, n_D^{22} 1.4132. It was prepd by nitration of the parent with HNO_3/Ac_2O (Ref 2)

Refs: 1) Beil – not found 2) G. Desseigne, BullSocChim **1946**, 98–9 & CA **41**, 2001 (1947) 3) E. Frommel et al, HelvPhysiol-PharmacolActa **10**, 438–47(1952) & CA **47**, 5545(1953)

1-Ethoxyethyl Hydroperoxide,

$C_2H_5O.CH(O_2H).CH_3$; mw 106.12, bp 0° (2 torr), 62–64° (14 torr), n_D^{25} 1.4091, sp gr ^{25} 1.005. It was prepd by reacting ethyl vinyl ether with 48% hydrogen peroxide and sulfuric acid (Ref 3), by heating 1,1-diethoxyethane with 100% hydrogen peroxide at 70°(Ref 4) or by heating β-ethoxy ethylacetate with 100% hydrogen peroxide at 65°(Ref 4). Spectrophotometric determination (Ref 5)

Refs: 1) Beil **1**, [674] 2) A.M. Clover, JACS **44**, 1107–18(1922) & CA **16**, 1930(1922) 3) N.A. Milas, R.L. Peeler, Jr & O.L. Mageli, JACS **76**, 2322–5(1954) & CA **49**, 8091(1955) 4) A. Rieche & C. Bischoff, Ber **94**, 2722–6 (1961) & CA **56**, 4606a(1962) 5) W.C. Wolfe, AnalChem **34**, 1328–30(1962) & CA **58**, 1910 (1963)

3-β-Ethoxyethyl-1,2,4-triazole,

$CH_3.CH_2.O.CH_2.CH_2-\overline{C=N-NH-CH=N}$; mw 141.17, N 29.77%, bp 130°(0.5 torr), n_D^{25} 1.4785. It was prepd from thiosemicarbazide and β-ethoxypropionyl chloride, followed by nitrous acid oxidation of the intermediate thio (Ref 2)

4-β-Ethoxyethyl-4H-1,2,4-triazole,

$CH_3.CH_2.O.CH_2.CH_2-\overline{N-CH=N-N=CH}$; mw 141.17, N 29.77%, bp 145–9°(0.05 torr). It was prepd via the thiosemicarbazide route (Ref 3), as in preceding item

Refs: 1) Beil – not found 2) C. Ainsworth & R.G. Jones, JACS **76**, 5654(1954) & CA **49**, 13979(1955) 3) S.A. Greenheld, M.C. Seidel & W.C. vMeyer, GerP 1943915(1970) & CA **72**, 100713(1970)

1-Ethoxy-2[1′-hydroxy-1′,4′-dimethyl-pentene-4′]-ethine,

$CH_2:C(CH_3).CH_2.CH_2.C(CH_3)(OH).C{\equiv}C.OC_2H_5$; mw 182.26, bp 82–84°(1 torr), sp gr 0.9258, n_D^{25} 1.4635. This product was prepd by Arens & Modderman (Ref 2) in the course of their work on synthesis of a lower homilog of citral and of pseudoionane. Severe explosions were produced during the distillation of the crude product if it had been stored for 1 night or longer. No explns accompanied distillation of freshly prepd batches

Refs: 1) Beil – not found 2) J.F. Arens & P. Modderman, ProcKoninklNederlandAkad-Wetenschap **54B**, 236–39(1951) & CA **46**, 1966(1952)

1-Ethoxymethyl-3,5-dinitro-1,3,5-triazacyclohexane or **1-(Ethoxymethyl) hexahydro-3,5-dinitro-5-triazine,**

$EtO.CH_2.\overline{N.CH_2.N(NO_2)CH_2.N(NO_2)CH_2}$; mw 235.20, N 11.90%($NO_2$), mp 118–19°. It was prepd by nitration of hexamine dinitrate at –28 to –60° followed by reaction with cold alc; readily nitrated to the 1,3,5-trinitro compound. It was also prepd by reacting alc with 1-acetoxymethyl-3,5-dinitro-1,3,5-triazacyclohexane (Ref 2)

Refs: 1) Beil – not found 2) W.J. Chute et al, CanJRes **27B**, 503–19(1949) & CA **43**, 9074

(1949) 3) K.W. Dunning & W.J. Dunning, JChemSoc **1950**, 2920–24 & CA **45**, 6642(1951)

3-(Ethoxymethyl)-hexahydro-1,5-dinitro-1,3,5(1H)-triazepine,

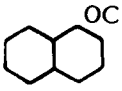
$CH_3.CH_2.O.CH_2-N-CH_2.N(NO_2)CH_2.CH_2.N(NO_2)CH_2$; mw 249.23, N 11.24%($NO_2$), mp 166.2–166.4°. It was prepd from 1-acetoxymethyl-3,6-dinitro-1,3,6-triazacycloheptane by boiling with alc (Ref 2) and by mixing N,N'-dinitroethylene-diamine with formalin, then adding alc and ammonium acetate (Ref 3)

Refs: 1) Beil – not found 2) G.S. Myers & G.F. Wright, CanJRes **27B**, 489–502(1949) & CA **43**, 9074(1949) 3) S.J. Strycker, USP 3522241(1970) & CA **73**, 66639(1970)

Ethoxy-MSX. Code name for 7-Ethoxy-2,4,6-trinitro-2,4,6-triazaheptane, described under 7-Ethoxy-2,4,6-triazaheptane and Derivatives

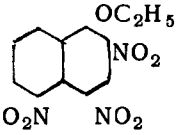
Ethoxynaphthalene and Derivatives

1-Ethoxynaphthalene,



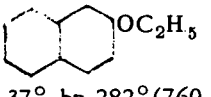
mw 173.04, mp 5.5°, bp 276.4°(760 torr), 160° (19 torr), 106°(2 torr), sp gr ^{25} 1.058, $n_D^{15.7}$ 1.6035, $n_D^{32.8}$ 1.5951. It was prepd by refluxing α-naphthol with ethyl bromide in alcoholic KOH (Ref 1)

1-Ethoxy-2,4,5-trinitronaphthalene,



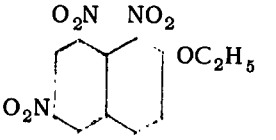
mw 307.22, N 13.68%, yel ndls, mp 148°, sol in HAc. It was prepd by nitration of the parent at 0°(Ref 2)

2-Ethoxynaphthalene,



mw 173.04, pltlts, mp 37°, bp 282°(760 torr), 142°(12 torr), sp gr ^{25} 1.0615, $n_D^{35.6}$ 1.5975, sol in alc, eth, petr eth, carbon disulfide & toluene. It was prepd by same method as the 1-ethoxy-deriv

2-Ethoxy-1,6,8-trinitronaphthalene,



mw 307.22, N 13.68%, mp 186°. It was prepd by nitration of 2-ethoxynaphthalene or displacement by NaOEt on the 2-Cl or 2-Br- nitrated naphthalenes

Refs: 1) Beil **6**, 606, 620, (306) & [578]; **6**, 641, (312) & [598] 2) W. Staedel, Ber **14**, 900(1881) 3) W. Staedel, Ann **217**, 170–71(1883)

2-Ethoxy-7,8-dinitroso-trinitronaphthalene,

mw 365.22, N 19.32%, OB to CO_2 –81.1%, yel ndls, mp 167°, sol in benz. It was prepd from 7,8-dinitroso-x,x,x-trinitro-2-oxy-naphthalene Ag salt and EtI (Ref 2). The Ref gives the following structure, –C(7)=N–O–O–N=C(8)–, for the dinitroso section of the new compound

Refs: 1) Beil **8**, 300 2) R. Nietzki & T. Knapp, Ber **30**, 1121(1897)

Ethoxypentane and Derivatives

Ethoxypentane, $C_2H_5O.C_5H_{11}$; mw 116.21, bp 119–20°. It was prepd by reacting ethoxyacetyl chloride and butyl zinc iodide in benz

1,1,1-Trinitro-2-ethoxypentane (TNEP), $C_3H_7.CH.C(NO_2)_3$

OC_2H_5 ; mw 251.20, N 16.71%, OB to CO_2 –86.1%. This expl compd has been prepd by reaction of butyraldehyde, ethanol & nitroform: $C_3H_7CHO + HC(NO_2)_3 + C_2H_5OH \rightleftharpoons$ TNEP $+ H_2O$; and by the simpler acetal reaction: $CH_3CH_2CH_2CH(OC_2H_5)_2 + HC(NO_2)_3 \rightleftharpoons$ TNEP $+ C_2H_5OH$ (Ref 3)

Refs: 1) Beil **1**, (193) 2) E. Blaise & L. Picard, AnnCh [8] **25**, 259(1912) 3) C.S. Rondestvedt Jr, Univ of Michigan Final Rept (Proj 2265)(May 1956)

Ethoxyphenylaminotetrazole. See under AMINOETHOXYPHENYLTETRAZOLES in Vol 1 of Encycl, p A202-L

Ethoxyphenylarsinic Acid and Derivatives

4-Ethoxyphenylarsinic Acid,

$C_2H_5.O.C_6H_4.AsO(OH)_2$; mw 246.10, wh prisms, mp 185°(–H_2O). It was prepd by diazotization of p-phenetidine and Na_3AsO_3 in alcoholic soln (Ref 2)

3,5-Dinitro-4-ethoxyphenylarsinic Acid,

$C_2H_5.O.C_6H_2(NO_2)_2.AsO(OH)_2$; mw 336.09, N 8.33%, OB to CO_2 –71.4%; wh crysts, mp – explodes at 275°, sol in alc & hot water. It was prepd by nitration of the parent (Refs 2 & 3)

Refs: 1) Beil **16**, 874 & [467]-Parent; [470]-Dinitro 2) M.P. deLange, Rec **45**, 52(1926) & CA **20**, 1983(1926)(Parent and dinitro deriv) 3) J.F. Morgan et al, JACS **69**, 930–2(1947) & CA **41**, 4464(1947)(Prepd by nitrating parent; recrystallized material is yel)

Ethoxyphenyltetrazole and Derivatives

p-Ethoxyphenyltetrazole,

p-EtO–C_6H_4–N–N=N–N=CH; mw 190.21, N 29.46%. It may be considered as the parent compd of its Azido deriv, although not used to prep it

p-Ethoxyphenyltetrazoloazide or **Azido-p-ethoxyphenyltetrazole,**

p-EtO–C_6H_4–N–N=N–N=C(N_3); mw 231.22, N 42.41%, mp 72°(dec 140°), pltlts (from alc), ndls (from water), sol in alc & eth. It was prepd from p-ethoxyphenyl thiourea, NaN_3 and $PbCO_3$ to get the *aminotetrazole* (mp 197°), followed by conversion of the amine to an azide; the intermediate *nitrosamine* has mp 117°(dec) and the *hydrazine*, mp 158°(dec)

Refs: 1) Beil – not found 2) R. Stollé, JPraktChem **134**, 282–3 & 303–4(1932) & CA **26**, 5565(1932)

Ethoxyphenylurethane and Derivatives

p-Ethoxyphenylurethane,

p-EtO–C_6H_4–$NHCO_2Et$; mw 209.25, ndls or pltlts (from alc), mp 93.5–94°, sol in alc, eth, chlf, benz & HAc. It was prepd by reacting ethyl chloroformate with p-phenetidine in alc or benz (Ref 1)

2,3,6-Trinitro-4-ethoxyphenyl urethane,

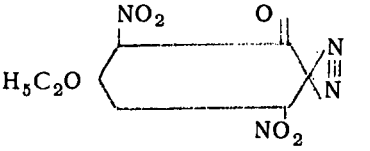
p-EtO–$C_6H(NO_2)_3$–$NHCO_2Et$; mw 345.05, N 12.18%(NO_2), OB to CO_2 –88.1%, yel ndls (from alc), mp 211–212°(dec), 218–19°(Ref 3), sol in alc, eth, benz, HAc & acetone. It was prepd by boiling the parent with nitric acid (Ref 2) or by warming N-nitro-N-ethyl-N′-(2,3,6-trinitro-4-ethoxyphenyl)-urea in alc (Ref 3)

Refs: 1) Beil **13**, 480(parent); 533 & [295](trinitro) 2) H. Köhler, JPrChim[2] **29**, 257(1884)(parent), 279(trinitro)(1884) 3) H.F.J. Lorang, RecTravChim **46**, 643(1927) 4) H. vPelchrzmi, ChemZentr **1924**, I, 654 & CA **18**, 3180(1924)(2-nitro deriv)

Ethoxyquinonediazole and Derivatives

Ethoxyquinonediazide, $C_8H_8N_2O_2$, may be considered as the parent compd of its nitro derivs although not used to prep them:

4-Ethoxy-3,6-dinitro-o-benzoquinonediazide(1),



mw 254.16, N 22.04%, OB to CO_2 –81.9%; or prisms (from acet ac), mp 186°. It was prepd by diazotizing 2,3,5-trinitro-4-aminophenetole (Ref 2)

3(?)-Ethoxy-5,6-dinitro-o-quinonediazide,

$C_8H_6N_4O_6$, mw 254.16, N 22.04%, OB to CO_2 –81.9%, yel pltlts (from alc), mp 166°(dec), expl on rapid heating; sol in alc, HAc & EtAc. It was prepd by heating the 3,5,6-trinitro-benzoquinone-(1,2)-diazide-(2) in alc (Ref 3)

Refs: 1) Beil **16**, (366)(Dinitro-4-ethoxy) 2) F. Reverdin & L. Fürstenberg, JPrChem [2] **88**, 327(1913) & CA **7**, 3314(1913) 3) G. Heller et al, JPrChem **129**, 246–47 (1931) & CA **25**, 2129(1931)

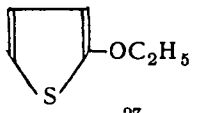
5-Ethoxytetrazole, C_2H_5OC–N–N–N–N

(location of two double bonds and one hydrogen atom within the ring varies with desmotropic isomerism), mw 114.11, N 49.10%, ndls (from benz), mp 98°, sol in w, eth, alc & EtAc. It was prepd from HN_3 and dimethyl azodicarbonate. *Silver salt* expl on heating

Refs: 1) Beil **26**, [237] 2) R. Stollé & G. Adam, Ber **57**, 1658(1924)

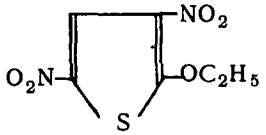
Ethoxythiophene and Derivatives

2-Ethoxythiophene,



mw 128.19, bp 56°(13 torr), sp gr ^{27} 1.075, n_D^{27} 1.5116. It was prepd by reacting 2-iodothiophene with CuO and NaOEt in EtOH under reflux (Ref 3)

3,5-Dinitro-2-ethoxy-thiophene,



mw 218.19, N 12.84%, yel ndls, mp 94–5°. It was prepd by boiling ethanol with the 2-Cl compd and sodium bicarbonate. The 2-Cl was made by a two stage nitration of 2-chlorothiophene (Ref 2)

Refs: 1) Beil – not found 2) C.D. Hurd & K.L. Kreuz, JACS **74**, 2969(1952) & CA **48**, 9355(1954) 3) J. Sicé, JACS **75**, 3699(1953) & CA **48**, 10726(1954)

Ethoxytriazaheptane and Derivatives

1-Ethoxy-2,4,6-triazaheptane,

$H_3C.NH.CH_2.NH.CH_2.NH.CH_2(OC_2H_5)$; mw 147.22, N 28.54%. Not found in open literature

Refs: 1) Beil – not found 2) CA – not found

1-Ethoxy-2,4,6-trinitro-2,4,6-triazaheptane, code named **MSX,**

$H_3C.N(NO_2).CH_2.N(NO_2).CH_2.N(NO_2).CH_2(OC_2H_5)$; mw 282.215, N 14.89%, OB to CO_2 –68.1%. It was prepd from reaction of 3-Me-1,5-dinitro-5-triazine with nitric acid, trituration with Et_2O and refluxing with fused NaAc in HAc (Ref 2); no props given. Ref 3 mentions a UV spectrum

Refs: 1) Beil – not found 2) F. Chapman, P.G. Owston & D. Woodcock, JChemSoc **1949**, 1647–48 & CA **44**, 1413(1950) 3) R.N. Jones & G.D. Thorn, CanJRes **27B**, 831(1949) & CA **44**, 2848(1950)

Ethylacardite. See Acardite III or N′-Ethyl-N,N-diphenylurea in Vol 1, p A8-L

Ethylacetamide and Derivatives

Ethylacetamide, $CH_3.CO.NH–CH_2.CH_3$; mw 87.12, N 16.08%, bp 205°, 104–5°(18 torr), sp gr$^{4.5}$ 0.942, sol in water & alc. It was prepd from ethyl acetate and ethylamine, ethylisocyanate and acetic acid (Ref 1)

N-(2,2-Dinitroethyl)acetamide,

$CH_3CO.NH.CH_2.CH(NO_2)_2$; mw 177.12, N 15.81%, OB to CO_2 –58.7%, mp 58–9°. It was prepd by removal of one $–NO_2$ from N-(2,2,2-trinitroethyl)-acetamide with KI & CH_3OH followed by H_2SO_4 at 0°C (Ref 7)

N-(2,2,2-Trinitroethyl)acetamide,

$H_3C.CO.NH.CH_2.C(NO_2)_3$; mw 222.115, N 25.23%, OB to CO_2 –28.8%, wh ndls, mp 82–84° (Refs 2 & 3), 88–90°(Ref 5), 91–92°(Ref 6). It was prepd from nitroform and N-methylolacetamide (Refs 5 & 6). Impact sensitivity to a 2.5kg hammer is 35cm (Ref 6)

N-Nitro-N-(2-dinitroethyl)acetamide,

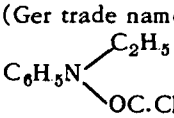
$CH_3CO.N(NO_2)CH_2.CH(NO_2)_2$; mw 222.15, N 25.23%, OB to CO_2 –28.8%, mp 73–74°. It was prepd from N-(2,2,2-trinitroethyl)acetamide by removal of one $–NO_2$ followed by nitration of the amide N (Ref 7)

N-(2,2,2-Trinitroethyl)azidoacetamide,

$N_3CH_2.CO.NH.CH_2.C(NO_2)_3$; mw 263.13, N 37.26%, OB to CO_2 –21.3%. Not known in open literature. See Ref 4

Refs: 1) Beil **4**, 109 & [601](parent) 2) USRubberCo, Summary Rept, March 1948, p 3 3) USRubberCo, Quarterly Rept **No 6**(1949), pp 6–7 and **No 8**, pp 7–8 & 25 4) USRubberCo, Quarterly Rept **No 25** on Contrs No 10129 & 12663, Nov 1, 1953 to Feb 1, 1954, p 9 5) NitroglycerinAktiebolaget, BritP 813477 (1959) & CA **53**, 19885a(1959) 6) P.O. Tawney, USP 3038010(1962) & CA **57**, 7512c (1962) 7) R.G. Gafurov, A.G. Korepin & L.T. Eremenko, IzvAkadNaukSSSR, SerKhim **1970**, (2), 442–3 & CA **73**, 3406e(1970)

n-Ethylacetanilide (Äthylacetanilid, in Ger) (Ger trade name *Mannol*),



mw 163.21, N 8.59%; white

to slightly reddish crysts; mp 54.5°, more difficultly subliming than camphor, but easier than Centralite; bp 258°(731 torr) for pure and 246–248° for technical; fl p 124°C; sol in water & ether. It is fairly stable against acids and alkalies in cold, but can be saponified on heating for several hours with concd alcoholic KOH. It can be prepd by reacting ethyl iodide with sodium acetanilide in xylene

Since it is a good gelatinizer for Collodion Cotton, it was used in German expls and proplnts, replacing camphor (Ref 4)

4-Nitro-N-ethylacetanilide (Nitromannol), $O_2N.C_6H_4.N(C_2H_5)(OC.CH_3)$; mw 208.22, N 13.45%, wh pltlts, mp 117.5°, sol in alc & benz. It was prepd by nitration of the parent with nitric acid of sp gr 1.52 (Ref 2) or with nitric acid (sp gr 1.48) in concd sulfuric acid (Ref 3)

Refs: 1) Beil **12**, 246, (194) & [143] 1a) Beil **12**, 720 (4-Nitro deriv) 2) A. Weller, Ber **16**, 31 (1883) 3) E. Noelting & A. Collin, Ber **17**, 267 (1884) 4) Kast-Metz, "Chemische Untersuchung der Spreng- und Zündstoffe", Vieweg & Sohn, Braunschweig (1944), pp 160–61 5) CondChemDict (1961), 456-R

ETHYL ACETATE AND DERIVATIVES

Ethyl Acetate (Et Acet) or *Acetic Ester (Acetic Ether)* (Essigester in Ger), $CH_3COO.C_2H_5$; mw 88.10; colorless, pleasant smelling liquid; sp gr 0.901 at 20/4°; fr p −83.6°(−82.4°), bp 77.2°, fl p −2°(26°F); coeff of expansion per 1°C 0.00133; heat of combstn at C_p 536.9 kcal/mole; heat of formation −110.72; sl sol in w (8.15g/100ml at 15°); miscible with alc or ether; 100ml of ethyl acetate dissolves about 3.5ml of w at 25°. It can be prepd by heating acetic acid with ethanol in presence of sulfuric acid, followed by distillation

Ethyl Acetate is irritating to mucous surfaces particularly the eyes, gums and respiratory passages, and is a mild narcotic. Therefore, its recommended maximum allowable concentration is 400 parts per million. Repeated and chronic exposures should not be allowed (Ref 6)

Ethyl Acetate is a good solvent for all Nitrocelluloses and has been used by A. Nobel in prepn of Gelatin Dynamites and then in prepn of smokeless proplnts. It can be used in mixts with acetone as in prepn of French "Poudre T", but use of straight acetone is unadvisable because it produces brittle colloids (Ref 3, p 87)

Possibilities of transporting NG in ethyl acetate are discussed in Ref 4. Prepn, props, uses and shipping are discussed in Ref 5

Refs: 1) Beil **2**, 125, (54) & [129] 2) M. Berthelot & L.P. de Saint-Gilles, ACh [3] **66**, 5 (1862) (First prepn listed in Beil) 3) E. de Barry Barnett, "Explosives", Van Nostrand, NY (1919), 87 4) A. Le Roux, MP **35**, 65 (1953) 5) CondChemDict (1961), 456–57 6) Federal Specification TT-E-751c, April 13, 1967, "Ethyl Acetate, Technical" (Intended use: as an ingredient in cellulose lacquers and lacquer thinners) 7) Kirk & Othmer, 2nd Edn, **8**, 378 (1965) 8) N.I. Sax, 3rd ed, p 732 (1968)

Ethyl Acetate, Technical. US Armed Forces Specification Requirements and Tests are covered by Federal Specification **TT-E-751c**, April 13, 1967, superseding IntFedSpec TT-E-00751b (Army–GL), Nov 30, 1965

EtAcet is usually delivered in 55-gallon metal drums, for noncorrosive materials, such as covered by FedSpec PPP–D–729

Specification requirements and tests for EtAcet are:

1. Appearance – shall be clear and free from sediment and suspended matter

2. Odor – shall have characteristic "banana oil" odor and shall leave no residual odor when tested accdg to FedTestMethodStd No 141a, Method 4401, Sept 1, 1965

Procedure: Note the odor immediately upon opening the container. In addition, dip a strip of heavy filter paper (Whatman No 2 or equal) into container with EtAcet and hang the test strip on a suitable support. Place it in a draft-free area away from any hoods and under atm condition to allow EtAcet to dry. Examine the test strip at suitable intervals for the normal "banana oil" odor. The presence of any additional odor shall be regarded as indicating the presence of an adulterant

3. Color – not darker than No 10 on the platinum-cobalt scale as prescribed by ASTM Method D1209. This method is described in this Vol, under "Ether, Diethyl: US Armed Forces Specification Requirements and Tests," pp E186 to E188

4. Specific Gravity – min 0.883 & max 0.888 at 20°/20°(68°/68°F) shall be detd by any convenient method that is accurate to the third place (such as by pycnometer, described in Vol 3 of Encycl, pp D69 to D71, under Density and Specific Gravity)

5. Acidity – free acid as acetic acid 0.01% by wt. This is equivalent to 0.093mg of KOH per gram sample. Use Procedure 4.3.3 of Spec MIL-E-463B described here under ETHANOL, Analytical Procedures

6. Water Content – 0.20% by wt. This limit insures that the material is miscible without turbidity with 19 volumes of 99% heptane at 20°. Determination is conducted by Karl Fischer Method, as described in ASTM D1364 and here as Spec MIL-E-463B, Procedure 4.3.7 under ETHANOL, Analytical Procedures

7. Distillation Range at 760mm – initial boiling point 70° and dry point 80°C. Use any standard distillation apparatus

8. Nonvolatile Matter – 0.005g per 100ml. Pipet 100ml of EtAcet into tared 150ml evaporating dish (previously heated in an oven at 105°±5° to constant wt and cooled in a desiccator), and evaporate the contents over a steam bath almost to dryness. Then heat the dish with contents in an oven at 105°±5° for 1 hour, cool in a desiccator and weigh. Return the dish with contents to the oven for 30 mins, cool and reweigh. Repeat, if necessary, until the wt is constant or within 0.1mg of the last weighing

9. Ester Content (as Ethyl Acetate) – min 85.0 & max 88.0%. It shall be detd in accordance with ASTM D1617–69, described in Annual Book of ASTM Standards, Part 20 (1972), 736–39

Note 1: The method described in ASTM D1617–69 applies not only to EtAcet, but also to other esters, such as N-butyl acetate, isopropyl acetate, acetate ester of ethylene glycol monoethyl ether, sec-butyl acetate, amyl acetate, dibutyl phthalate and isobutyl acetate

Note 2: Org chlorides, nitriles and amides interfere. Ketones and aldehydes interfere only slightly with this procedure

In this method the sample is reacted with a measured excess of aq KOH, using isopropanol as a mutual solvent, if required (but not for EtAcet). The amt of KOH consumed, which is detd by titrating the excess with std mineral acid, is a measure of the ester originally present. Since this detn is based on acidimetric titration, a suitable correction should be applied if the acidity of the sample exceeds the limit of spec

Apparatus:

a) Pressure Bottle – 200 to 350ml capacity made from heat-resistant glass. Bottles of this type, equipped with lever-type closures, can be obtd from the B. Preiser Co, Charleston, WVa

b) Container for Pressure Bottle. A metal safety enclosure with hinged top and perforated bottom, a strong synthetic fabric or canvas bag, or a safety shield can be used

c) Ampoule – 1 or 2ml capacity

d) Wei ghing Pipet – Lunge or similar

Note 3: It was found during WWII in the laboratory of Keystone Ordnance Works, Meadville, Pa that Berl Pipet and Modified Weighing Pipet were more convenient to work than Lange's (See Figs Et 11 and Et 12)

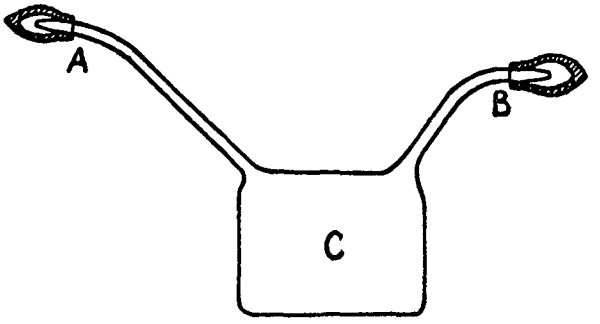


FIG Et11 – BERL PIPETTE

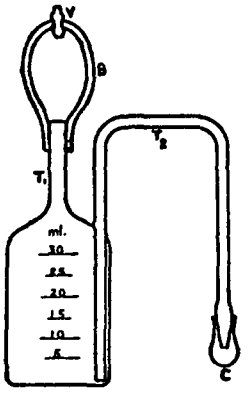


FIG Et12

e) Erlenmeyer Flasks – 250ml capacity, Pyrex with glass stoppers
f) Buret – 50ml capacity
g) Boiling Water Bath

Reagents:

Reagent grade chemicals, such as conforming to the ACS Specifications shall be used and water conforming to the Specifications for Reagent Water, ASTM Designation D1193

a) Hydrochloric Acid, Standard (0.5N). Prep and standardize against 1.0N KOH std reagent to 4 significant figures. Used when isopropanol is added as solvent (See operation c under Procedure)
b) Isopropanol – 99% (Not required for EtAcet)
c) Phenolphthalein Solution – 1g of phph in 100ml methanol
d) Potassium Hydroxide, Standard Solution (1.0N) – 66g of KOH pellets dissolved in reagent water and diluted to 1 liter (Do not use NaOH soln as a substitute)
e) Sulfuric Acid, Standard (0.5N). Prep and standardize to 4 significant figures. Used when isopropanol is not added as solvent (See opn c, under Procedure)

Procedure:

a) Prepare two Erlenmeyers for blanks and two for samples tested in cold. For testing at 98° prep two pressure bottles
b) Into each of the flasks and pressure bottles (except those intended for blanks) carefully introduce 25ml of 1.0N KOH soln, using the same calibrated pipet for each transfer
c) This opn prescribes addn to each flask the amt of isopropanol prescribed in Table I of ASTM D1617–69, but such addn is not required for ethyl acetate or acetate ester of ethylene glycol monoethyl ether
d) Stopper two of the flasks and two pressure bottles of opn b and set them aside for the blanks. Into each of the other two flasks and two bottles, introduce sealed ampoules, each contg not more than 0.016 mol of the ester, weighed to the nearest 0.1mg. In case of EtAcet, the wt must be not higher than 88.10 x 0.016 = 1.4096g. The flasks and bottles must be strong enough to withstand the next opn of breaking ampoules
e) Introduce to each of the two sample flasks and two bottles several of fire-polished 8-mm glass rods, stopper and shake the flasks and bottles vigorously to break the ampoules
f) *Reaction at 98°C.* Enclose each of the four pressure bottles securely in a protective container (listed as opn b, under Apparatus) to restrain fragments of glass should the bottle rupture. Place the samples and blanks as close together as possible in the boiling water bath maintained at least at 98°C for 30 minutes. The amt of water in the bath must be sufficient to just cover the liquid in the bottles. Remove the bottles from the bath and allow to cool to RT. Then remove the bottles from the protective containers and uncap the bottles carefully to release any pressure. Continue as prescribed in opn h
g) *Reaction at Room Temperature.* Allow the two Erlenmeyers with blanks and two with samples to stand for 30 minutes at RT and then proceed as in opn h
h) If a white ppt develops in the sample bottles (heated at 98°) or in sample Erlenmeyers left for 30 mins at RT, add sufficient amt of water to dissolve the ppt. This amt shall be the same for each bottle or Erlenmeyer, contg samples and blanks. Add 6 to 8 drops of the phph indicator to each flask or bottle and titrate with std 0.5N sulfuric acid soln just to the disappearance of the pink color
i) Measure the temp of the acid and, if it is not the same as it was at the time the reagent was standardized, apply a correction to the normality. Use a $\Delta N/\Delta T$ of 0.00014 per °C in making the correction
j) *Calculation*

Calculate the percentage of ethyl acetate as follows:

$$\% \text{ EtAcet} = \frac{(B-A)\,C \times F}{W} \times 100$$

where:

A = mls of 0.5N sulfuric acid required for titration of sample, as described under opn h
B = average mls of 0.5N sulfuric acid required for titration of the blanks
C = normality of acid corrected for temperature
F = factor for EtAcet specified in Table II as 0.0881, and
W = grams of sample used in each bottle or Erlenmeyer

Note 4: If the acidity of the ester exceeds 0.01% by wt as free acetic acid (See opn 5 of FedSpec TT-E-751C), a suitable correction must be applied to the ester value

k) Report the results to the nearest 0.1%. Duplicate detns which agree within 0.38% absolute are acceptable for averaging

Ethyl Acetate, Use in French Propellants and Its Determination in Them

Accdg to Dalbert & Tranchant (Ref 1), ethyl acetate has been used as a volatile gelatinizer in some French smokeless proplnts, such as **T & Tbis** of the Pouderie de Sevran

Estimation of EtAcet in these proplnts can be conducted by the following method:

Treat a 20g sample of proplnt with 120ml of 36° Bé NaOH(40% aqueous soln) and 280ml of distd water. This decomposes EtAcet into ethanol and Na acetate. Distill off about 150ml of liquid into a receptacle cooled in ice and filter the distillate. Add to the distillate, with cooling and swirling, about 40ml of concd sulfuric acid and exactly 30ml of the soln contg 48.453g potassium dichromate per liter of sulfuric acid. Heat on a water bath for 20 minutes in order to oxidize the ethanol, cool, transfer to a 250ml volumetric flask and adjust to volume by adding some sulfuric acid. Pipet out 25ml of soln and titrate the excess of dichromate by means of KI and 0.1N sodium thiosulfate soln. Then titrate a blank prepd by mixing the same ingredients as above except that the 150ml distillate is replaced by 150ml of distd water

Calculation:

$$\text{Weight of EtAcet in Propellant} = 0.0352 \times 30 \times \frac{N-n}{N}$$

where:

N = ml of sodium thiosulfate soln required for blank, and

n = ml of the same soln required for sample (Ref 1)

In Ref 2 is described analysis of industrial EtAcet liable to contain water, ethanol and ethyl formate with traces of acidity, whereas in Ref 3 is described a precise method for detn of acidity in EtAcet

In Ref 4 is described a rapid method for detn of the amount of EtAcet & ethanol in their mixtures with water, accdg to the density and water content as detd by the Fischer Method

Refs: 1) R. Dalbert & J. Tranchant, MP **30**, 343–45(1948) 2) G. Bourjol, MP **32**, 291–300 (1950) 3) J. Burlot & G. Bourjol, MP **32**, 301–04(1950) 4) G. Bourjol, MP **37**, 459(1955)

Ethyl Acetate, Azido- and Nitrated Derivatives

Ethyl acetate Azide, Ethyl azidoacetic Acid or **Azidoacetic Acid Ethyl Ester,** $N_3CH_2.CO.OC_2H_5$; mw 129.12, N 36.41%, chlf odor, bp 74–75°(23 torr), 62°(12 torr), 45°(2 torr), sp gr^{20} 1.127, sp gr^{25} 1.118, n_D^{25} 1.4341. It was prepd in 1908 (Refs 2 & 3) by refluxing ethyl iodoacetate with AgN_3 or ethyl chloroacetate with NaN_3

Refs: 1) Beil **2**, 229, (101) & [208] 2) T. Curtius, A Darapsky & A. Bockmühl, Ber **41**, 352(1908) & CA **2**, 1429(1908) 3) M.O. Forster & H.E. Fierz, JChemSoc **93**, 79(1908) & CA **2**, 853(1908) 4) J.C. Philip, JChemSoc **93**, 919 (1908) 5) E.A. Shott-L'vova & Y.K. Syrkin, DoklAkadNaukSSSR **87**, 639–41(1952) & CA **47**, 6203(1953) (Dipole moment) 6) W.F. Huber, JACS **77**, 112–16(1955) & CA **50**, 804(1956) (Original prepn) 7) W.R. Carpenter, Appl-Spectry **17**(3), 70–2(1963) & CA **59**, 4684(1963) (IR spectrum) 8) F. Minisci & R. Gallo, Chim-Ind(Milan) **47**(2), 178–80(1965) & CA **63**, 5553 (1965) (New prepn) 9) H. Koneig & W. Reif, GerP 1280866(1968) & CA **70**, 37467(1969) (New prepn) 10) A.J. Papa, USP 3429879 (1969) & CA **70**, 87819(1969) (New prepn)

Ethyl acetate Diazide (Bistriazo-essigsäure-äthylester in Ger), $(N_3)_2CH.CO.OC_2H_5$; mw 170.13%, N 49.4%, OB to CO_2 –84.7%, colorless, odorless, liquid, bp 70–72°(about 2 torr), sp gr^{18} 1.222, sp $gr^{24.9}$ 1.220, $n_D^{24.9}$ 1.4640. It was prepd in 1908 by refluxing ethyl dichloroacetate with NaN_3 (Ref 3). It tended to expl in contact with concd sulfuric acid. It was considered too dangerous to explore further

Refs: 1) Beil **2**, 230 & (101) 2) J.C. Philip, JChemSoc **93**, 919–20(1908) 3) M.O. Forster, H.E. Fierz & W.P. Joshua, JChemSoc **93**, 1073 (1908) 4) Ibid, **97**, 130 & 1368(1910)

2-Azidoethyl-azidoacetate,

$N_3CH_2.CO.O.CH_2.CH_2N_3$; mw 170.13, N 49.4%, OB to CO_2 –84.7%, bp 115°(8.5 torr), 90°(1.1 torr), n_D^{30} 1.4798. It was prepd in 1908 by heating azidoacetyl chloride with 2-azidoethanol at 100° (Ref 2), and in 1964 by reacting chloroethyl chloroacetate with NaN_3 in dimethylformamide (Ref 3). It detonates mildly when placed on a hot plate; apparently reacts vigorously with concd sulfuric acid and hot KOH soln

Refs: 1) Beil **2**, 229 2) M.O. Forster & R. Müller, JChemSoc **95**, 201(1909) & CA **3**, 1526(1909) 3) J.A. Durden Jr, H.A. Stansbury & W.H. Catlette, JChemEngData **9**(2), 228–31 (1964) & CA **60**, 15859(1964)

Ethylnitroacetate, $O_2N.CH_2.CO.OC_2H_5$; mw 133.10, N 10.53%; colorless liq, sp gr 1.199 at 20/4°, n_D^{20} 1.4247, bp 105–07° at 25mm; v sl sol in w, sol in alc, insol in eth. It can be prepd by the action of KOH on nitromalonic acid diethyl ester

Refs: 1) Beil **2**, 225, (100) & [207] 2) A. Wahl, CR **132**, 1053(1901)

Ethyldinitroacetate, $(O_2N)_2CH.CO_2.C_2H_5$; mw 178.10, N 15.72%, OB to CO_2 –44.9%, colorless oil, bp 50–54°(0.05 torr, Ref 4) and 40°(0.5 torr, Ref 5), n_D^{20} 1.4336, sp gr^0 1.369. It was prepd by ambient nitration of monoethylmalinate (Ref 2)

Refs: 1) Beil **2**, 228, (100) 2) L. Bouveault & A. Wall, CR **136**, 159(1903) 3) L.W. Kissinger & H.E. Ungnade, JOrgChem **23**, 1340–2(1958) & CA **53**, 15951(1959) (Prepn) 4) C.A. Parker, USP 2925433(1960) & CA **54**, 11480(1960)(Prepn) 5) Ibid(3) **24**, 666–8(1959) & CA **54**, 18335(1960) (Prepn)

Ethyltrinitroacetate, $(O_2N)_3C.CO.OC_2H_5$; mw 223.10, N 18.83%, OB to CO_2 –17.9%

Refs: 1) Beil, not found 2) CA, not found

2,2,2-Trinitroethyl Acetate, $CH_3.COO.CH_2.C(NO_2)_3$; mw 223.10, N 18.83%, OB to CO_2 –17.9%; colorless oil, fr p 26° (Ref 6), bp 85–95°(in vacuo) (Ref 3). Ref 2 gives 118° at 4mm; does not decomp appreciably when heated up to 140°; sl sol in w. It can be prepd by treating trinitroethanol with acetyl chloride

Trinitroethanol Acetate is stable at RT and insensitive to impact (does not deton by 5kg wt falling from one meter height)

It is a good gelatinizer for NC and could be used in manuf of smokeless proplnts or of dynamite type expls if its price were less expensive

Refs: 1) Beil, not found 2) N.S. Mavans & R.P. Zelinski, JACS **72**, 5329–30(1950) & CA **45**, 4642g(1951) 2a) M. Hill, NAVORD Rept **2245**(1951)(Prepn & props) 3) H. Ficheroulle & A. Gay-Lussac, MP **34**, 121–23 (1952) & CA **48**, 4838–39(1954) 4) S.S. Novikov, G.A. Shvekhgeimer & N.F. Pyatakov, IzvestAkadN, OtdKhimNauk **1961**, 375 & CA **55**, 19774(1961)(Prepd by heating the alcohol with ethoxyacetylene and HCl, bp_4 90°, n_D^{20} 1.4528, sp gr 1.4743 at 20°) 5) M.E. Hill, USP 3223725(1965) & CA **64**, 6570(1966)(Prepd by reacting the alcohol with acetyl chloride and a Friedel-Crafts catalyst in 85% yield) 6) N.D. Lebedeva, V.L. Ryadnenko & I.N. Kuznetsova, ZhFizKhim **1968**, 42(7), 1827 (Russ) & CA **69**, 90540(1968)(sp gr 1.4794 at 20°, n_D 1.4484 at 20°, bp_1 64.5°, mp 26°, heat of combustion 1982 [no units given in CA]) 7) L.T. Eremenko, N.G. Zhitomirskaya & G.V. Oreshko, IzvAkadNaukSSSR, SerKhim **1969**, (12), 2674–87(Russ) & CA **72**, 84439–41(1970) (IR spectrum)

Trinitroethylazidoacetate, $N_3.CH_2.COO.CH_2.C(NO_2)_3$, mw 264.11, N 15.91% ($NO_2$), OB to CO_2 –12.1%. Nothing in open literature. See Ref 3. It is an explosive

Refs: 1) Beil, not found 2) CA, not found 3) USRubberCo QuartRept No **25** on Contr Nos 10129 & 12663, Nov 1, 53 to Feb 1, 54(1954), p 8

Dinitroethyldinitroacetate, $(O_2N)_2.CH.COO.CH_2.CH(NO_2)_2$, mw 268.10, N 20.89%, OB to CO_2 0.0%. Not known in open literature

Refs: 1) Beil, not found 2) CA, not found

Trinitroethyldinitroacetate, $(O_2N)_2CH.COO.CH_2.C(NO_2)_3$; mw 313.0, N 22.36%, OB to CO_2 +12.8%; solid. Not known in open literature.

Refs: 1) Beil, not found 2) CA, not found

Trinitroethyltrinitroacetate, $(O_2N)_3C.COO.CH_2.C(NO_2)_3$, mw 358.09, N 23.47%, OB to CO_2 +22.3%. Not known in open literature

Refs: 1) Beil, not found 2) CA, not found

Ethylacetic Acid. See Butyric Acid in Vol 2 of Encycl, p B391ff

Ethylacetoacetic Acid and Derivatives

Ethylacetoacetic Acid, Ethylacetoacetate or *Acetoacetic Ester,* $CH_3CO.CH_2.COO.C_2H_5$; mw 130.14; colorless liq of fruity odor, sp gr 1.025 at 20/4°, mp –45° (–80°), bp 180° at 755mm; sl sol in w; miscible with alc, eth & chlf. It can be prepd by the action of metallic Na on ethyl acetate with subsequent distillation. Used in org synthesis,

pharmaceuticals, dopes, plastics, etc
Refs: 1) Beil **3**, 632, (223) & [415] 2) Cond-ChemDict (1961), 457-L 3) Beil **7**, [940]

Ethylacetoacetic Acid Diazide, Ethyl-α,α-bistriazoacetoacetate or **Diazidoethylacetoacetate,** $CH_3CO.C(N_3)_2.COO.C_2H_5$; mw 212.16, N 39.68%, fuming liq, bp 81–82°(0.9 torr). It was prepd by action of NaN_3 on the α,α-dichloroanalogue in aq alc (Ref 2)

It is an explosive compd; in one of the attempts to det its %N, an expln resulted
Refs: 1) Beil **3**, (233) 2) M.O. Forster & Newman, JChemSoc **97**, 1367–68(1910)

Ethylacetone. Same as Methyl-n-propylketone or 2-Pentanone, described in Beil **1**, 676 and in CondChemDict (1961)

Ethylacetonitrolic Acid and Derivatives
Ethylacetonitrolic Acid or *Ethylnitroacetaldoxime,* $CH_3C(NO_2)=NO.C_2H_5$, mw 132.12, N 21.20%. Not known in open literature
Refs: 1) Beil – not found 2) CA – not found

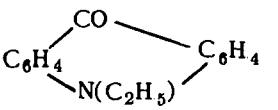
1,1-Dinitroethyl-acetonitrolic Acid; N(1,1-Dinitroethyl)-1-nitroacetaldoxime or **2,5,5-Trinitro-3-aza-4-oxa-hexene-2,** $CH_3C(NO_2)=N-O-C(NO_2)_2CH_3$ or, possibly, $CH_3C(NO_2)=N(O)C(NO_2)_2CH_3$; mw 222.11, N 25.22%, OB to CO_2 –28.8%, mp 121.2–121.6°, heat of combustion, H_C, 544.8kcal/mol. It was prepd from 1,1-dinitroethane and its ammonium salt (Ref 2)
Refs: 1) Beil, not found 2) J.S. Belew, C.E. Grabiel & L.B. Clapp, JACS **77**, 1110–14(1955) & CA **50**, 1648(1956) 3) J.A. Young et al, IndEngChem **48**, 1375–8(1956) & CA **50**, 16274 (1956) 4) S.S. Novikov, I.S. Korsakova & K.K. Babievskii, IzvestAkadNaukSSSR, OtdelKhimNauk **1959**, 1480–1 & CA **54**, 1284(1960)(Prepn)

Ethylacetylene and Derivatives
Ethylacetylene or *1-Butyne,* $C_2H_5.C{:}CH$; mw 54.09; colorless, flammable gas, available in commerce in liquefied form; sp gr 0.669 at 0/0°, fr p –125.8°, bp 8.7°(8.3°); fl p (Tag open cup) <20°F; insol in w; sol in alc & eth. It can be prepd by action of potassium on 2,2-dichlorobutane, or treatment of 2-bromobutene-1 with alcoholic KOH at 100°(Ref 2). It is used as specialty fuel and as an intermediate
Refs: 1) Beil **1**, 248, (107), [233] & {924} 2) M.E. Reboul, CR **113**, 591(1891) 3) CondChemDict (1961),457-R

1-Nitroethylacetylene, $C_2H_5C{\equiv}C.NO_2$; mw 99.09, N 14.14%, red oil, expl on heating in vacuo or in air, polymerizes on standing. It was prepd by mixing 1-bromo-1-nitro-1-butene with methylamine
Refs: 1) Beil – not found 2) J. Loevenich, J. Koch & U. Pucknat, Ber **63B**, 636–46(1930) & CA **24**, 3211(1930)

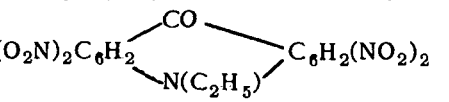
Ethylacridine and Derivatives
N-Ethylacridine, mw 209.29. Not found in literature. A **nitric acid salt** is mentioned in Ref 3. It should be possible to reduce N-ethylacridone (See following entry)
Refs: 1) Beil, not found 2) CA, not found 3) V. Zanker et al, ZPhysickChem (Frankfurt) **48**(3–4), 179–95(1966) & CA **65**, 2092(1966)

N-Ethylacridone,



mw 223.28, gr-yel prisms (from alc), mp 159°; sol in alc. It was prepd by heating potassium acridone with C_2H_5I at 130–40°(Ref 2)
Refs: 1) Beil **21**, 336 2) C. Graebe & K. Lagodzinski, Ber **25**, 1736(1892) 3) B.I. Mikhant'ev & V.A. Sklyarov, ZhurObshchei-Khim **26**, 784–5(1956) & CA **50**, 14760(1956) (Prepn)

N-Ethyl-2,4,5,7-tetranitroacridone,



mw 403.27, N 13.89%, mp 282°. It was prepd from heating ethylamine with o,o′-dimethoxy-m,m,m′,m′-tetranitrobenzophenone
Refs: 1) Beil, not found 2) C.W. Pohlmann, RecTravChim **55**, 737–52(1936) & CA **30**, 7111(1936)

Ethylacrylate and Derivatives
Ethylacrylate, $CH_2{:}CH.COO.C_2H_5$; mw 100.11; colorless liq, sp gr 0.925 at 15°, fr p –72°(–75°), bp 100–01°(99.8°); sl sol in w (9% by wt);

readily polymerizes. It can be prepd by interaction betw ethylenecyanohydrin and ethanol in dil sulfuric acid, or by oxo reaction of acetylene, CO and ethanol in the presence of Ni or Co catalyst; used as a chemical intermediate and in prepn of polymers and acrylic paints
Refs: 1) Beil **2**, 399, (186) & [386] 2) W. Caspary & B. Tollens, Ann **167**, 248(1873) 3) CondChemDict (1961), 457-R

Ethyl-2-nitroacrylate, $O_2N.CH{=}CH.CO_2.C_2H_5$; mw 145.11, mp 100–2°. It was prepd by adding $Na^+CH_2NO_2^-$ to $C_2H_5.O_2C.CH{=}CH.CH.O$, converting the resulting alcohol to a chloride with PCl_3, and splitting out an internal $-CH.ClCH_2-$ group with NaAc
Refs: 1) Beil, not found 2) L.A. Yanovskaya, R.N. Stepanova & V.F. Kucherov, IzvAkadNauk-SSSR, SerKhim **1964**(11), 2093–5 & CA **62**, 7630e (1965)

2-Nitroethylacrylate, $CH_2{=}CH.CO_2.CH_2.CH_2.NO_2$; mw 145.11, bp 100°(5 torr) and **ca** 50°(ca 0.2 torr), sp gr 1.204 at 25°, n_D 1.4510 at 25°. It was prepd by distillation from a mixture of methylacrylate, 2-nitroethanol, hydroquinone, toluenesulfonic acid and xylene (Ref 2)
Refs: 1) Beil, not found 2) N.S. Marans & R.P. Zelinski, J ACS **72**, 2125–6(1950) & CA **44**, 8858(1950) 3) H.J. Roy Jr, USP 2710830(1955) & CA **50**, 5723(1956)(Purif) 4) J.W. Lawrence & H.F. Bluhm, USP 3179644 (1965) & CA **63**, 426(1965)(Prepn)

2-Nitratoethylacrylate, $CH_2{=}CH.CO_2.CH_2.CH_2.ONO$ mw 161.11, bp 100–3°(8 torr), sp gr ^{25} 1.246, n_D^{25} 1.4500. It was prepd by heating acrylyl chloride with 2-nitratoethanol in the presence of cuprous chloride
Refs: 1) Beil, not found 2) N.S. Marans & R.P. Zelinski, JACS **72**, 5330–1(1950) & CA **45**, 4648(1951)

Trinitroethylacrylate, $CH_2{:}CH.COO.CH_2.C(NO_2)_3$; mw 235.11, N 32.83%; OB –30.6%, bp 80°(2 torr), n_D^{20} 1.4640, dy^{20} 1.4492. It was prepd by heating together acrylyl chloride and 2-(trinitro)ethanol (Ref 1). It has impact sensitivity of 45 with 2kg weight (Ref 4)
Refs: 1) Beil, not found 2) H. Feuer, H.H. Hass & R.D. Lowrey, JOrgChem **25**, 2070–1 (1960) & CA **55**, 14300c(1961) 3) J. Ville, MP **42**, 21–7(1960) & CA **55**, 18109(1961) 4) R.H. Saunders, USP 2994714(1961) & CA **55**, 25758g(1961)

Ethyl Alcohol. See ETHANOL in this Vol, p E154 ff

Ethylaldehyde (Ethanal or Ethylidine Oxide). See ACETALDEHYDE in Vol 1, pp A14 to A15

Ethylamine. See AMINOETHANE in Vol 1, pp A199 to A200

Ethylamine-Azide. See Vol 1, p A199

N-Aminoethylacetamide and Derivatives
N-Aminoethylacetamide,
$CH_3.CO.NH.CH_2.CH_2.NH_2$, mw 102.14; bp 135–40°(2 torr). It was prepd by heating ethylenediamine with ethyl acetate (Ref 2)

N-(2-Nitraminoethyl) -acetamide,
$CH_3.CO.NH.CH_2.CH_2.NH.NO_2$; mw 147.14, N 28.56%, mp 134.5–135.5°. It was prepd by treatment of 1-acetyl-3-nitro-2-imidazolidinone with dil NaOH, followed by acidification (Ref 3)
Refs: 1) Beil, not found 2) K.W. Rosensnund, USP 1926015(1933) & CA **27**, 5339(1933) 3) M.W. Kirkwood & G.F. Wright, JOrgChem **18**, 629–42(1953) & CA **48**, 6968(1954)

Ethylaminoanisole and Derivatives
2-Ethylaminoanisole or *Ethylanisidine,*
$C_2H_5.NH.C_6H_4.OCH_3$; mw 151.20, oil; bp 117° at 3mm, 228–9° at 728mm. It may be prepd by heating at 59° a mixture of o-anisidine and ethyl bromide
Ref: Beil **13**, 364

Dinitroethylaminoanisoles,
$C_2H_5.NH.C_6H_2(NO_2)_2.OCH_3$; mw 240.19, N 17.49%
3,5-Dinitro-2-ethylaminoanisole, mp 123°; may be prepd from 2,3,5-trinitroanisole and ethylamine
Ref: Beil **13**, 393
4,6-Dinitro-3-ethylaminoanisole, mp 148°; may be prepd from 5-bromo-2,4-dinitroanisole and

ethylamine
Ref: Beil **13**, 424

3,5-Dinitroethyl-2-nitraminoanisole, $O_2N.N(C_2H_5).C_6H_2.(NO_2)_2.OCH_3$; mw 286.20, N 14.68%($NO_2$); mp 67°; may be prepd by treating 3,5-dinitro-2-ethylaminoanisole with nitric acid (d 1.52)
Refs: 1) Beil **13**, 394 2) J.J. Blanksma, Rec **23**, 113(1904)

Ethyl p-aminobenzoate or **Benzocaine**(Anesthesine), $C_6H_4.NH_2.COO.C_2H_5$; mw 165.19; wh crysts (from alc), mp 91–2°; v sl sol in w; sol in dil acids; less sol in chlf, eth & alc; exhibits local anesthetic props when placed on the tongue. It can be prepd by ethylation of p-nitrobenzoic acid, followed by reduction; used in medicine
Refs: 1) Beil **14**, 422, (567) & [248]
2) CondChemDict (1961), 458-R

Ethyl p-aminonitrobenzoates. $H_2N.C_6H_3.(NO_2).CO_2.C_2H_5$; mw 210.18, N 13.33%
Ethyl 4-amino-2-nitrobenzoate; lt yel ndls, mp 130°; sol in alc, eth; insol in w; may be prepd from hydrazinehydrate and ethyl 2,4-dinitrobenzoate in abs alc or from 4-amino-2-nitrobenzoic acid and ethyl iodide in alc potash or alc sulfuric acid
Ref: Beil **14**, 439 & (583)
Ethyl 4-amino-3-nitrobenzoate; mp 136°; insol in petr eth, sol in usual org solvents; may be prepd by refluxing ethyl 4-bromo-3-nitrobenzoate and alc NH_3 at 150° for 3 hrs; by esterification of 4-amino-3-nitrobenzoic acid with abs alc in sulfuric acid; or by refluxing ethyl 4-ethoxy-3-nitrobenzoate and alc NH_3
Ref: Beil **14**, 441

Ethyl p-aminodinitrobenzoates, $H_2N.C_6H_2.(NO_2)_2.CO_2.C_2H_5$; mw 255.18, N 16.49%
Ethyl 4-amino-3,5-dinitrobenzoate; gold-glimmering leaflets, mp 114°. It may be prepd by esterification of the acid in alc HCl
Ref: Beil **14**, 458

Ethylaminocellulose. See Aminoethylcellulose in Vol 1, p A203-R

Ethylaminocellulose Nitrate and Perchlorate are described in Vol 1, p A204

Ethylamino Compounds. See Vol 1, pp A204 to A209

Ethylaminoethanol and Derivatives
β-Ethylaminoethanol or *Ethylethanolamine,* $C_2H_5.NH.CH_2.CH_2OH$; mw 89.14, colorless oil, sp gr 0.914 at 20/4°; fr p ?, bp 167–69° at 751mm, fl p 160°F; v sol in w, alc & eth; can be prepd from ethyl bromide and excess 2-amino-ethanol at 50–60°; used as solvent and for prepn of nitrated derivs, such as Et-NENA
Refs: 1) Beil **4**, 282, [727] & {667} 2) J.S. Pierce et al, JACS **64**, 1692(1942) 3) CondChemDict (1961), 470-R (Ethylethanolamine; props but no prepn)

Ethylaminoethanol Nitrate, $C_2H_5.NH.CH_2.CH_2.ONO_2$; mw 134.13, N 20.88%; crysts (from methylethylketone), mp 68°; may be prepd by treating 2-ethylaminoethanol with concd nitric acid at –5°
Refs: 1) Beil **4**, {668} 2) C. Barbière, BullFr [5] **11**, 470, 472(1944)

1-(N-Ethyl)-nitramino-2-ethanol Nitrate; N-(β-Nitroxyethyl)-ethylnitramine or **N-(2-Nitratoethyl)-ethylnitramine,** code named **Et-NENA,** $(C_2H_5).N(NO_2).CH_2.CH_2.ONO_2$; mw 179.13, N 23.46%, OB to CO_2 –67.0; pale yel oil, sp gr 1.32 at 25/4°, n_D 1.479 at 25°; fr p 4.0–5.5°, bp ?. It was prepd by Blomquist & Fiedorek (Refs 2 & 3) by nitration of ethylaminoethanol. For this, ethylaminoethanol was added dropwise, with stirring, to 98% nitric acid, while maintaining the temperature at 10°C; then the resulting mixture was transferred to a dropping funnel and added dropwise, with stirring, to 95% acetic anhydride contg some acetyl chloride

Following reactions took place:

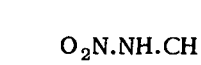
$$(C_2H_5).NH.CH_2.CH_2OH \xrightarrow{HNO_3} (C_2H_5)\left(\begin{matrix}NH_2^+\\NO_3^-\end{matrix}\right)\text{-}CH_2.CH_2.ONO_2 \xrightarrow{Ac_2O} (C_2H_5).N(NO_2).CH_2.CH_2.ONO_2$$

An improved procedure called "two-stage liquid-phase" is described in Ref 4

Et-NENA may be considered as a possible plasticizer for NC in double-base propellants
Refs: 1) Beil, not found 2) A.T. Blomquist & F.T. Fiedorek, OSRD Rept **4134**(1944) & PB Rept **18867**(1944), pp 44–5 & 118 3) Ibid, USP 2485855(1949), pp 5 & 11–12 and CA **44**, 3516–17(1950) 4) Ibid, USP 2678946(1954) & CA **49**, 4704(1955)

Ethylaminoethylguanidine and Derivatives
Ethylaminoethylguanidine,
$H_2N.CH_2.NH.C(:NC_2H_5)NH_2$; mw 130.19, N 43.04%
Refs: 1) Beil, not found 2) CA, not found
2-Ethyl-1-(2-nitraminoethyl)-3-nitroguanidine,
$O_2N.NH.CH_2.CH_2.NH.C(:NC_2H_5)NH.NO_2$; mw 220.19. N 38.19%; may be prepared from 1-nitro-2-nitramino-2-imidazoline dissolved in aq $EtNH_2$ and adjusting the pH to 1 at a temperature of less than 20°
Refs: 1) Beil, not found 2) A.F. McKay and C. Sandorfy, CanJChem **31**, 42–7(1953) & CA **47**, 6765(1953) 3) A.F. McKay & W.G. Hatton, JACS **75**, 963–65(1953) & CA **48**, 2049(1954)

Ethylaminoethyl-pseudourea and Derivatives
Ethylaminoethyl-pseudourea, mw 131.17
Refs: 1) Beil, not found 2) CA, not found
2-Ethyl-1-(2-nitraminoethyl)-3-nitropseudourea,

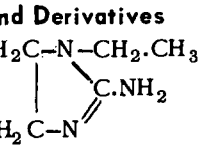


$O_2N.NH.CH_2.CH_2.NH.CNH.NO_2$; mw 221.18, N 31.67%, mp 133.6–134°; may be prepd by refluxing 1-nitro-2-nitramino-2-imidazoline with EtOH for 19 hours
Refs: 1) Beil, not found 2) A.F. McKay & C. Sandorfy, CanJChem **31**, 42–7(1953) & CA **47**, 6765(1953)

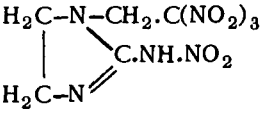
3(β-Ethylaminoethyl)-α-sym-triazole Dipicrate. See in Vol 1, p A208-R

Ethylaminoguanidine. See Aminoethylguanidine in Vol 1, p A205-R

Ethylaminoimidazoline and Derivatives
Ethylaminoimidazoline,



mw 113.16, N 37.13%; may be considered as a parent compd of Trinitroethylnitraminoimidazoline although not used for its prepn
1-(2,2,2-Trinitroethyl)-2-nitramino-2,3-imidazoline.



mw 293.16, N 33.45%; long, wh ndls, mp 168–69°(decomp); burns rapidly and readily detonates; may be prepd from 2-nitramino-1,3-diaza-2-cyclopentane & nitroform
Refs: 1) Beil, not found 2) USRubberCo, 5th Rept (1948–49), p 33 & Summary Rept on Contr NOrd 10129, p 3 3) H.A. Hageman, USP 3043848(1962) & CA **57**, 14045(1962)

Ethylaminonylon and Its Perchlorate. See under Aminoethylnylon in Vol 1, p A205

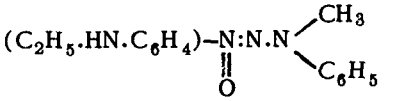
Ethylaminophenols and Derivatives
N-Ethylaminophenols or *N-Ethylhydroxyanilines,*
$C_2H_5.NH.C_6H_4.OH$; mw 137.18. All three isomers: o-, m-, and p- are known and described in the literature
Refs: 1) Beil **13**, 364(ortho) 2) Beil **13**, 408 (meta) 3) Beil **13**, 443, (150) & [231](para)
Dinitro-N-ethylaminophenols,
$C_2H_5.NH.C_6H_2(NO_2)_2OH$; mw 277.17, N 15.16%($NO_2$)
3,5-Dinitro-3-ethylaminophenol. It may be prepd by nitration of N-ethylbenzoxazoline with nitric acid, giving the 4,6-dinitro derivative. Alkaline hydrolysis gives the 3,5-dinitro phenol derivative
4,6-Dinitro-3-ethylaminophenol, yel ndls, mp 128–29°; sol in hot w; may be prepd from 4,6-dinitro-3-ethylaminophenotol by saponification with hot alcoholic potash
Refs: 1) Beil **13**, (138) 2) G. Wagner & S. Leistner, Pharmazie **26**, 280–2(1971) & CA **75**, 35852(1970)

Trinitro-N-ethylaminophenol,
$C_2H_5.NH.C_6H(NO_2)_3OH$; mw 272.17, N 20.29%
2,4,6-Trinitro-3-ethylaminophenol; mp 115°; may be prepd by exchange with $EtNH_2$ on either

2,3,4,6-tetranitrophenol or 3-chloro-2,4,6-trinitrophenol in alcohol
Ref: Beil **13**, (140)

Trinitro-N-ethylnitraminophenol,
$C_2H_5.N(NO_2).C_6H(NO_2)_3OH$; mw 317.18, N 22.08%
2,4,6-Trinitro-3-ethylnitraminophenol; mp 105–6°; may be prepd by treating N-ethyl-N-(2,3,4,6-tetranitrophenyl)nitramine with boiling water or by nitration of the trinitroethylaminophenol above with nitric acid (d 1.52)
Refs: 1) Beil **13**, (140) 2) J.J. Blanksma, Rec **21**, 260 (1902)

[N′-(4-Ethylaminophenyl), N′-oxy-N³-methyl-N³-phenyl]-triazene or **[N³-Methyl-N³-phenyl-N′-oxy-N′-(4-ethylaminophenyl)]-triazene**
{Called in Ger: (3-Methyl-3-phenyl-1-[4-äthylaminphenyl]-triazenoxyd-(1)};



mw 270.34, N 73%; yel ndls (from alc), mp 97°; deflgr on rapid heating; can be prepd from p-nitroso-N-ethylaniline hydrochloride [See Beil **7**, (345)] and α-methylphenylhydrazine in acetic acid soln, under cooling
Refs: 1) Beil **16**, (414) 2) O. Fischer, JPraktChem [2], **92**, 65 (1915) & JChemSoc **108** (Abstract 1), 908 (1915)

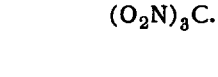
Ethylaminopolyurethane. See Aminoethylpolyurethane in Vol 1, p A205-R

Ethylaminopolyvinyl Alcohol and Its Chloride and Perchlorate. See under Aminoethylpolyvinyl Alcohol in Vol 1, p A205-R

Ethylaminoprotein. See Aminoethylprotein in Vol 1, p A206-L

Ethylaminopropylureas. See Aminoethylpropylureas in Vol 1, p A206-L

N-Ethylaminotetrazoles. See AMINOETHYLTETRAZOLES AND DERIVATIVES in Vol 1, pp A206 & A207 and
5-(Trinitro-N-ethylamino)-tetrazole,



N———N; mw 248.12, N 45.16%, NO_2 N 16.94%, OB to CO_2 −13%; crysts, mp 145–46°, explodes at higher temps; sol in methanol or in 1:1 mixture of nitromethane/2-nitroethane

Accdg to description given in ADL Rept (Ref 3): to the soln of 2g (0.01 mole) aminotetrazole hydrate in 20ml warm methanol was added 2ml (0.027 mole) of 40% formaldehyde and 3g (0.02 mole) of nitroform and the mixture refluxed for 3 hours. The resulting red soln was filtered (to remove a small amt of solid) and filtrate diluted with 40ml of distd w. After standing for 2 days in an ice chest, the resulting ppt was collected on filter, dissolved in 20ml methanol and pptd by adding 40ml distd w. The yield was nearly quantitative

It is a very powerful explosive (about 161% TNT by Trauzl Test)
Refs: 1) Beil, not found 2) R. Stollé & Schiek, GerP 426343 (1924) 3) R.H. Saunders et al, Hercules Powd Co Monthly Progress Rept **13** (Sept 1949) (Contract NORD 9925) (Compd investigated for possible use as a primary expl, however, its water solubility & thermal stability were considered unsatisfactory. Its impact sensitivity with BurMinesApp with 2kg wt was 5cm and the 100°C Vacuum Stability Test value exceeded the capacity of the app in 48 hrs) 4) ADL, Synthesis HE's, 2nd Rept (1951), pp 55–6

N-Ethylaminotoluenes. See N-Ethyltoluidines in this Vol

Ethylaminotriazoles. See AMINOETHYLTRIAZOLES in Vol 1, pp A207 to A209-L

Ethylaminoureas. See Aminoethylureas in Vol 1, p A209

N-Ethylaminoxylenes or **Ethyldimethylanilines.** See N-Ethyloxylidines in this Vol

Ethylammonium Tetranitratozincate.
$(EtNH_3^+)_2\ Zn(NO_3)_4^{--}$; mw 405.84, N 20.71%;

mp 83–4°, decomp at 190–210°, explodes on rapid heating to 270°; deliquescent; sol in $MeNO_3$, acet, cyclohexanone, water and EtOH; insol in liq N_2O_4, C_6H_6, ether, dioxane, CCl_4 & $CHCl_3$; may be prepd from $Zn(NO_3)_2$ & $EtNH_3NO_3$ in liq N_2O_4 soln

Refs: 1) Beil, not found 2) C.C. Addison & N. Hodge, JCS **1954**, 1138–43 & CA **48**, 7477 (1954)

N-ETHYLANILINE AND DERIVATIVES

N-Ethylaniline; N-Phenylaminoethane or *Phenylethylamine*, $C_2H_5.NH.C_6H_5$; mw 121.18, N 11.56%; colorless liq, turning brown on exposure to light; sp gr 0.963 at 20/4°, fr p –65.8°, bp 205.5°; n_D 1.5559 at 20°; insol in w & petr eth; miscible with alc & eth. It can be prepd by heating aniline with ethanol in presence of sulfuric acid, followed by distillation (Ref 3)

It was used (accdg to Dr H. Walter) during WWII as one of the ingredients of German liquid rocket fuels

Small quantities of ethylaniline, usually present in Centralites, can be detd by polarographic method described by Sifre (Ref 2)

Refs: 1) Beil **12**, 159, (155) & [90] 2) G. Sifre, MP **35**, 382 (1953) 3) CondChemDict (1961), 458-R

Note: N-Ethylaniline should not be confused with ortho-, meta- and para-Ethylanilines, $C_2H_5.C_6H_4.NH_2$, described here under Ethylphenylamines, that are called also Aminoethylbenzenes

Nitro-N-ethylanilines, $C_8H_{10}N_2O_2$, mw 166.19, N 16.85%. The following isomers are described in the literature:

2-Nitro-N-ethylaniline, $C_2H_5.NH.C_6H_4(NO_2)$; reddish oil (Ref 1)

3-Nitro-N-ethylaniline, same as above; red-yel ndls, mp 59–60° (Ref 2)

4-Nitro-N-ethylaniline, same as above, lt-yel scales, mp 95–96° (Refs 3 & 5)

β-Nitroethyl-aniline, $(O_2N)CH_2.CH_2.NH.C_6H_5$; lflts, mp 37° (Ref 4)

Refs: 1) Beil **12**, 690 2) Beil **12**, 702 3) Beil **12**, 714, (350) & [386] 4) Beil **12**, (156) 5) P.C. Condil, IEC **41**, 1704–09 (1949) & CA **44**, 1430 (1950)

N-Nitro-N-ethyl-4-nitroaniline, $C_2H_5.N(NO_2).C_6H_4(NO_2)$; mw 211.17, N 19.90%; yel ndls, mp 90°

Ref: Beil **12**, 729

Dinitro-N-ethylanilines, $C_8H_9N_3O_4$; mw 211.17, N 19.90%

The following isomers are described in the literature:

2,4-Dinitro-N-ethylaniline, $C_2H_5.NH.C_6H_3(NO_2)_2$, yel or om crysts, mp 114–119° (Refs 1 & 4)

2,5-Dinitro-N-ethylaniline, same as above, red ndls, mp 120° (Ref 2)

3,5-Dinitro-N-ethylaniline, same as above, om crysts, mp 185–6° (Ref 3)

Refs: 1) Beil **12**, 750, (362) & [406] (2,4-Dinitro) 2) Beil **12**, (365) (2,5-Dinitro) 3) J.C. Roberts & K. Selby, JChemSoc, 2785–89 (1949) & CA **44**, 3922 (1950) (3,5-Dinitro) 4) W.A. Schroeder et al, IEC **42**, 539 (1950) & CA **44**, 4253 (1950)

Trinitro-N-ethylanilines, $C_8H_8N_4O_6$; mw 256.17, N 21.87%; the following isomers are known:

2,4,6-Trinitro-N-ethylaniline or **N-Ethyl-2,4,6-trinitroaniline** (called **Ethylpicramide** by van Romburgh), $C_2H_5.NH.C_6H_2(NO_2)_3$; yel crysts, mp 81.5°; explodes at higher temps; sl sol in w; sol in alc & very sol in acet & benz; numerical values of solubilities in various solvents are given by Desvergnes (Ref 3). It can be prepd from picrylchloride and ethylamine in alcoholic soln (Refs 1 & 2)

It was investigated in Germany during WWII (Ref 4) as a possible substitute for proplnts used in mortars. The development never came to a practical realization due to the unfavorable raw materials situation

Refs: 1) Beil **12**, 764, (368) & [421] 2) P. van Romburgh, Rec **2**, 107 (1883) 3) L. Desvergnes, MonitScient [5] **16**, 203 (1926) 4) R.E. Richardson et al, CIOS Rept **25-18** (1945), 28 5) W.A. Schroeder et al, IEC **42**, 539 (1950) & CA **44**, 4253 (1950)

x,x,x-Trinitro-N-ethylaniline, $C_2H_5.NH.C_6H_2(NO_2)_3$; pale-yel ndls (from alc or ethyl acetate), mp 178.5°. It was prepd by Desvergnes by heating N,N′-diethyl-N,N′-bis (x-trinitrophenyl)-urea in 70% sulfuric acid. It is an explosive

Refs: 1) Beil **12**, [426] 2) L. Desvergnes, RevChimInd **37**, 42 (1929)

N-Ethyl-2,4,6-trinitro-N-nitrosoaniline, $C_2H_5.N(NO).C_6H_2(NO_2)_3$; mw 285.18, N 24.56%; may be prepd by nitration of the nitrosamine of monoethylaniline with nitric and sulfuric acids. The compd is an explosive

Refs: 1) Beil, not found 2) T.J. Nolon et al, BritP 217614(1922) & CA **19**, 300(1925)

N-Ethyl-N,2,4,6-tetranitroaniline; (2,4,6-Trinitrophenylnitramino)ethane; N-Ethyl-N-nitro-2,4,6-trinitroaniline; Ethylpicrylnitramine; called by Blatt **2,4,6-Trinitrophenylethylnitramine** and code named **Ethyl Tetryl** (Called in Beil: N-Nitro-N-äthyl-2.4.6-trinitroanilin; N-Äthyl-N.2.4.6-tetranitroanilin; Äthyl-pikryl-nitramin or Teträthyl; French name **Tétréthyl**), $C_2H_5.N(NO_2).C_6H_2(NO_2)_3$; mw 301.18; N 23.26%, OB to CO_2 −61.1%; pale yel monoclinic prismatic crysts; sp gr 1.644 at 10°; mp 95–96°; explodes at higher temp; insol in w, sol in alc, benz, chlf and nitric acid (See Ref 6). It was prepd by van Romburgh (Refs 1 & 2) by treating with nitric acid mono- or di-ethylaniline. Blanksma (Refs 1 & 3) prepd it from N-ethyl-4-nitroaniline by nitration; can also be prepd by nitrating ethylphenylnitrosamine. It was prepd after WWII in France and its properties determined (Refs 9, 10 & 11)

It is an expl more powerful than TNT (111% or 115% as detd by Trauzl Test), but more sensitive to impact (FI 92% PA). Its detonation velocity is 6200m/sec at sp gr 1.10, vs 5000 for TNT at the same sp gr (Refs 7 & 8)

It was proposed as a booster charge in HE shells

Refs: 1) Beil **12**, 771, (371) & [426] 2) P. van Romburgh, Rec **2**, 111(1883) 3) J.J. Blanksma, Rec **22**, 272(1902) 4) E. Burkard, USP 1049665 & CA **7**, 893(1913)(Detonating fuse containing Trinitroethylaniline) 5) T.J. Nolan & H.W. Clapham, USP 1522025 & CA **19**, 733(1925)(Ethyl Tetryl from ethylphenylnitrosamine and MA) 6) L. Desvergnes, MonitScient [5] **16**, 207(1926)(Solubilities in various liquids) 7) A.H. Blatt, OSRD **2014**(1944)(2,4,6-Trinitrophenylethylnitramine) 8) ADL, PureExplCompds, Part **1**(1947), p 27 9) G. Desseigne, MP **31**, 43 (1949) 10) L. Médard, MP **31**, 180(1949) 11) G. Desseigne, MP **36**, 21–6(1954)

N-Ethylaniline-pentazidocuprate, $[C_8H_{12}N][Cu_2(N_3)_5]$; mw 459.37, N 48.79%; mp– explodes with a loud report at 187–89°; burns with crackling noise and detonates on impact; was prepd from $CuCl_2.2H_2O$ in methanol by adding ethylaniline and sodium azide in hot water

Refs: 1) Beil, not found 2) A. Cirulis & M. Straumanis, Ber **76B**, 825–30(1943) & CA **38**, 1972(1944)

N-Ethyl-N,2,3,4,6-pentanitroaniline, $C_2H_5.N(NO_2).C_6H(NO_2)_4$; mw 346.17, N 24.28%; crysts, mp 96°; sol in benz & toluene; may be prepd by treating 3,4-dinitro-N,N-diethylaniline and sodium nitrate in dilute nitric acid and nitrating the resulting product with nitric acid (d 1.49) and sulfuric acid

Refs: 1) Beil **12**, (372) 2) P. van Romburgh & J.H. Schepers, AkadAmsterdamVersl **22**, 299(1913) & CA **8**, 3656(1914)

o-, m- and p-Ethylanilines. See Ethylphenylamines or Aminoethylbenzenes in this Vol (Not to be confused with N-Ethylaniline, also known as Phenylaminoethane)

Ethylazahexanoate and Derivatives

Ethyl-2-azahexanoate, $CH_3.CH_2.CH_2.CH_2.NH.COO.CH_2.CH_3$; mw 145.20, N 9.65%. It may be considered as the parent compd of its pentanitro deriv although not used to prep it

Trinitroethyl-2,5,5-trinitro-2-azahexanoate, $CH_3.C(NO_2)_2.CH_2.CH_2.N(NO_2)COO.CH_2.C(NO_2)_3$; mw 315.20, N 31.11%; solid, mp 107°, d 1.73, hot bar ignition temp 221°, sensitivity – approx equal to Pentolite. It was prepd by nitrating the carbamate from 3,3-dinitrobutyl isocyanate & trinitroethanol

Refs: 1) Beil, not found 2) D.V. Sickman & W.F. Sager, NAVORD Rept **486**(1954)(Res & Devel in New Chem HE's)

Ethylazaurolic Acid. See Vol 1, p A517-R under AZAUROLIC ACIDS

Ethyl Azelate or Diethyl Azelate and Derivatives

Ethyl Azelate, $(CH_2)_7(COO.C_2H_5)_2$; mw 244.32, liquid, sp gr 0.973 at 20/4°, fr p −16°, bp 291–92°; insol in w; sol in alc or eth. It can be prepd by

esterification of the acid with ethanol catalyzed by H_2SO_4 or HCl gas and with water removal by azeotropic distillation with benz or toluene (Ref 1). Other props in Ref 2
Refs: 1) Beil **2**, 709, (290), [603] & {1787} 2) G.A. Schmidt & D.A. Shirley, JACS **71**, 3805(1949)

Bis(trinitroethyl)azelate,
$(O_2N)_3C.CH_2OC(CH_2)_7COCH_2C(NO_2)_3$; mw 514.36, N 15.97%, OB to CO_2 −59%; liq, fr p −10°; sol in methylene chloride-pentane; impact sensitivity 90cm (for 50% point on BM Machine). It was prepd by heating slowly at 65° azelaic acid chloride & trinitroethanol over a period of 4 hrs. The reaction mixt was allowed to stand overnight, washed with water and recrystd at low temp from methylene chloride-pentane. This compd may be useful as plasticizers or desensitizers of expls (Ref 3)
Refs: 1) Beil, not found 2) CA, not found 3) D.L. Kouba et al, Hercules Progress Rept on High Explosives (2 Nov 1951)(Navy Contract NOrd 11280, Task A)

Ethyl Azide. See Azidoethane under Ethane and Derivatives, in this Vol, p E142-R

Ethylazidoacetate. See under Ethyl Acetate in this Vol

Ethylazidobenzamide or **N(β-Azido-ethyl)-benzamide,** $C_6H_5.CO.NH.CH_2.CH_2(N_3)$; mw 190.21, N 29.46%; oil; explodes violently on contact with concd sulfuric acid. It can be prepd from benzoyl chloride and β-azido-ethylamine in the presence of NaOH (Refs 1 & 2)
Refs: 1) Beil **9**, (97) 2) M.O. Forster & S.H. Newman, JChemSoc **99**, 1279(1911)

Ethyl-α-azido-ethylacetoacetate or **α-Ethyl-α-triazoethylacetoacetate** (α-Azido-α-äthyl-acetessigsäure-äthylester, in Ger),
$CH_3.CO.C(N_3)(C_2H_5).COO.C_2H_5$
mw 199.20, N 21.09%; oil; sp gr 1.0536 at 19°; bp 84–5° at 0.19mm. It may be prepd by heating the α-chloro-α-triazoethylacetoacetate in alcohol with aq sodium azide
Refs: 1) Beil **3**, (241) 2) M.O. Forster & S.H. Newman, JChemSoc **97**, 1365(1910)

Ethylazidoformate, $(N_3)COO.C_2H_5$; mw 115.09, N 36.51%; oil; bp 25° at 2mm & sp gr 1.118. It was prepd by stirring ethyl chlorocarbonate with sodium azide (aqueous). It is an explosive
Refs: 1) Beil **3**, 129 & [101] 2) M.O. Forster & H.E. Fierz, JChemSoc **93**, 81(1908)

Ethylazidomalonic Acid Diamide,
$C_2H_5.C(N_3)(CO.NH_2)_2$; mw 171.16, N 40.92%; mp 167°. It was prepd by shaking the ester with concd aq amine. The ester was prepd from ethyl bromomalonic ester and sodium azide by refluxing eight hours in 50% alcohol [bp 83.5° at 0.7mm, sp gr 1.1161(16°/16°)]
Refs: 1) Beil **2**, (276) 2) M.O. Forster & R. Müller, JChemSoc **97**, 134(1910)

Ethylbenzamide and Derivatives
N-Ethylbenzamide, $C_6H_5.CO.NH.C_2H_5$; mw 149.19, N 9.39%; n dls (from w), mp 70–1°, bp 298–300°; sl sol in hot w. It can be prepd in a number of ways, as indicated in Ref
Ref: Beil **9**, 202, (97)

N-[β-Azidoethyl]-benzamide,
$C_6H_5.CO.NH.CH_2.CH_2.N_3$, mw 190.21, N 29.46%; oil which decomposes violently on contact with concd sulfuric acid. It can be prepd from benzoyl chloride and β-azidoethylamine in the presence of NaOH
Refs: 1) Beil **9**, (97) 2) M.O. Forster and S.H. Newman, JCS **99**, 1279(1911)

3-Nitroethylbenzamide, $3NO_2.C_6H_4.CO.NH.C_2H_5$; mw 194.18, N 14.42%; mp 143°, bp 310–15°. It may be prepd from ammonium benzoate by heat-

ing (explodes), 3-Nitrobenzoyl chloride and ammonia, anhydride of 3-Nitrobenzoic and acetic acids and ammonia, 3-Nitrobenzoicbromamide and sodium ethoxide, and benzamide and abs nitric acid

4-Nitroethylbenzamide, $4\text{-}NO_2.C_6H_4.CO.NH.C_2H_5$; mp 151°; sol in alc, insol in w. It may be prepd from 4-Nitrobenzoyl chloride and $EtNH_2$

Refs: 1) Beil **9**, {1710} 2) H. Wenker, JACS **60**, 1081 (1938)

ETHYLBENZENE AND DERIVATIVES

Ethylbenzene or *Phenylethane*, $C_2H_5.C_6H_5$; mw 106.16; colorless liq, resembling methylbenzene (toluene), sp gr 0.867 at 20/4°, n_D 1.4959 at 20°, fr p −95.0°, bp 136.2°, fl p 85°F (29.5°C); sp heat 0.41cal/g, viscosity 0.64 centipoise at 25°; heat of combstn at C_v 108.9kcal/mole, heat of vaporization 81.1cal/g at bp; very sl sol in w (0.01% at 15°); v sol in alc, eth, benz & chlf; insol in Amm hydroxide. It can be prepd by heating benzene and ethylene in presence of Al chloride with subsequent distillation, or by fractionation directly from the mixed xylene stream in petroleum refining. Purification is by rectification. It is used in org synthesis, as solvent & diluent and for prepn of explosive azidonitro-, dinitro-, trinitro- & higher nitrated derivatives

Refs: 1) Beil **5**, 351, (175), [274] &{776} 2) CondChemDict (1961), 459-L 3) Kirk & Othmer **19**, 55 (1969); & CA **22**, 467 (1970)

Ethylbenzene Azide or **Azidoethylbenzene**, $C_2H_5.C_6H_4.(N_3)$; mw 147.17, N 28.54%. Refs were found only for α- and β-azidophenylethanes. No refs to o-, m- or p-azidophenylethanes were found

1-Azido-1-phenylethane, $N_3C(CH_3)H.C_6H_5$; liq, sp gr 1.0321 at 25/4°, n_D 1.5233 at 25°; bp 114° at 50mm, 83° at 20mm. It may be prepd by treating 1-chloroethyl benzene with sodium azide in aq methanol

Refs: 1) Beil **5**, {807} 2) P. Levene et al, JBiolChem **120**, 777 (1937) & CA **32**, 484–86 (1938) 3) F. Heron & E.D. Hughes, JCS **1969**, 795–9

2-Azido-1-phenylethane, $N_3CH_2.CH_2.C_6H_5$; oil, n_D 1.5308 at 25°; bp 121–25° at 20mm, 52–4° at 0.65mm. It may be prepd by treating $PhCH_2.CH_2.Br$ with sodium azide or with 1,1,3,3-tetramethylguanidiniumazide (Ref 3)

Refs: 1) P.A.S. Smith & B.B. Brown, JACS **73**, 2435–7 (1951) & CA **46**, 494 (1952) 2) J.H. Boyer & J. Hamer, JACS **77**, 951–4 (1955) & CA **50**, 1826 (1956) 3) A.J. Papa, USP 3429879 (1969) & CA **70**, 87819 (1969)

Ethylbenzene, Nitrated Derivatives were patented by the late Dr Hale of PicArsn as colloiding agents for NC in manuf of smokeless propellants

Ref: G.C. Hale, USP 1964826 (1934) & CA **28**, 5242 (1934)

1-Ethyl-mononitrobenzenes, $C_2H_5.C_6H_4.NO_2$, mw 151.16, N 9.27%, OB to CO_2 −195.3%

When ethylbenzene is nitrated, a mixture of ortho- and para-mononitrobenzenes in the proportion 2 to 1 is usually obtd. They are suitable as gelatinizers for NC

o-(or 2-) Nitro-1-ethylbenzene, colorless oil, sp gr 1.126 at 24.5°, fr p −23°, bp 227–28°; insol in w; v sol in alc; sol in eth (Ref 1); Q_c^v = 1048.06kcal/mole and Q_f^v = 11.75kcal/mole (Ref 3)

m-(or 3-) Nitro-1-ethylbenzene, sp gr 1.1345 at 0°, bp 242–43°. It may be prepd from 4-amino-3-nitroethylbenzene and isoamylnitrate. (Ref 2)

p-(or 4-) Nitro-1-ethylbenzene, sp gr 1.124 at 25°, fr p −32°, bp 245–46°; insol in w; v sol in alc or ether (Ref 1); Q_c^v = 1046.40kcal/mole and Q_f^v = 13.45kcal/mole (Ref 3)

Refs: 1) Beil **5**, 358, (178), [279] & {803} 2) Beil **5**, 358 3) L. Médard & M. Thomas, MP **39**, 197, 207 (1957)

α-Azido-β-nitroethylbenzene, $C_6H_5.CH(N_3)CH.NO_2$; mw 191.17, N 29.31%. It may be prepd from 1-nitro-2-phenylethene and sodium azide in acetic acid. It is violently decompd in concd sulfuric acid at RT

Refs: 1) Beil, not found 2) J.H. Boyer, JACS **73**, 5248–52 (1951) & CA **47**, 490 (1953)

4-Nitro(2-nitroethyl)benzene, $NO_2.CH_2.CH_2.C_6H_4.(NO_2)$; mw 196.16, N 14.28%, solid (from acet ac), mp 163–64°. It may be prepd from 2-nitro-2-(p-nitrobenzyl)-1,3-indandione and Na in ethanol

Ref: L. Zalukajevs & E. Vanags, ZhurObshchei-Khim **26**, 3115–19 (1956) & CA **51**, 8668 (1957)

1-Ethyl-2,4-dinitrobenzene or **2,4-Dinitro-1-ethylbenzene (DNEtB)**, $C_2H_5.C_6H_3(NO_2)_2$; mw 196.16, N 14.28%, OB to CO_2 −130.05%; yel oil liq, sp gr 1.3171 at 25°, n_D 1.5655 at 25°, fr p ?, bp 195.5° at 33mm; v sl sol in w; soly in 95% alc 9.34g in 100g at 4° and 22.70g at 25°; sol in ether. It is a mild expl which possesses the property of gelatinizing NC; has been used as a flash-reducing agent in smokeless proplnts in lieu of DNT (Refs 1–4). Tavernier (Ref 5) gives its Q_c^v =5145cal/g at 300°K and Q_f^v = 65cal/g. It can be prepd by a one- or two-stage nitration of ethylbenzene with nearly quantitative yields. Trial nitrations conducted at PicArsn have shown that the mixed acids must be weaker than those used for the nitration of toluene to the dinitro-stage, otherwise excessive oxidation of the ethyl group takes place

One-stage nitration of commercial ethylbenzene, conducted at PicArsn by Varrato (Ref 2) yielded an oil of fr p betw −12 & −16.5°, consisting mostly of DNEtB with N content about 14.3%. Although this product was found to be a better gelatinizing agent for NC than DNT, it was not desirable to use it because its N content was lower than that for DNT (N content about 15.4%), which would result in lower potential proplnts prepd with DNEtB. By pushing the nitration a little further, it was possible to obtain a product with N content approacing that of DNT (N content about 15.4%) and still to have an oil which was a good gelatinizer. This oil contd about 35% of Trinitroethylbenzene, while the rest (65%) consisted of DNEtB and impurities

When it was desired to prepare proplnts with higher potential than those with DNT, nitration went even further, such as to prepare a mixture of N content 16.3%, which contd about 64% of TNEtB and was still oily. Even if nitration of commercial EtB went to completion and the resulting product consisted mostly of TNEtB, it was still nearly liquid at RT (mp ca 21.5°) and a good gelatinizer for NC. Its N content was 17.25% vs 18.5% for commercial TNT (Ref 2)

Note: Accdg to remark made by Mr H.A. Aaronson (Refs 3 & 4) while he was still working at PicArsn and developed a process for nitration described below, a number of experimental proplnts were prepd at the Arsenal using DNEtB and its mixts with TNEtB as gelatinizers in lieu of DNT. He does not think that the question of N content is as important as the yields, which are poor for TNEtB and good for DNEtB

Plant Procedure for Preparation of 2,4-Dinitroethylbenzene

1) 50 lbs of EtB and 130 lbs of "cycle acid" were placed in a standard stainless steel nitrator with coils for heating and cooling and a double-paddle agitator

Note: "Cycle acid", which is weaker than mixed acid is used for starting the nitration in order not to allow the reaction to become too violent and to be able to more easily control the temp during the early stages of nitration. The compn of the "cycle acid" is similar to that used in the mononitration of toluene, such as H_2SO_4 53.5, HNO_3 7.2, Nitroso 15.0 & nitro bodies 1.0%, which was listed by Clift & Fedoroff in "A Manual for Explosives Laboratories", Vol 1, Chap III, p 2(1942)

2) While maintzining the temperature at 55°C and the agitation at 75RPM, 220 lbs of mixed acid (H_2SO_4 59.8 & HNO_3 22.1%) were slowly added. Total time of mononitration was about 1 hour, 15 mins

3) The mixture was allowed to settle for 6 mins to form a sharp line of separation, and the spent acid (which contd 6–10% nitric acid & 0.5–0.7% nitrobodies and separated at the lower part of the nitrator) was run off, until about 130 lbs of it remained in the nitrator

4) After this the agitation was set at 100RPM and the temp raised to about 80°C. Mixed acid (220 lbs) of the same compn as above was slowly added and the nitration continued for 1 hr, 45 mins at 80–83°C

5) The mass was then cooled to 40°, allowed to settle for 6 mins and the spent acid (which contd about 7–10% nitric acid and 1.3–1.5% nitrobodies) was run out

6) The remaining crude DNEtB was washed to neutrality with several changes of water (Ref 4)

Refs: 1) Beil **5**, 360, (178), [280] & {805} 2) P. Varrato, PicArsnTechRept **R91**, Problem 502.6–17/41 (1930) 3) H.A. Aaronson, PicArsnChemLabRept **53071**, April 1938 4) H.A. Aaronson, PATR **969** (June 1939) 5) P.F. Macy, "Explosive Plasticizers for Nitrocellulose", PATR **1638** (Feb 1947) 6) P. Tavernier, MP **38**, 308 & 329 (1956) & CA **50**, 3763 (1956)

2,4-Dinitroethylbenzene, US Specification Requirements and Tests were given in Joint Army-Navy Spec **JAN-D-685**, 30 Sept, 1948. We are including the Spec here although 2,4-DNEB is not used in current US proplnts as a replacement for DNT

1. Appearance. Clear, amber colored liquid, as determined by visual inspection

2. Specific Gravity. 1.31±0.01 at 15.5°/15.5°, as detd by Westphal Balance or by pycnometers (See Vol 3 of Encycl, pp D68 to D71, under Density and Specific Gravity)

3. Refractive Index. 1.563±0.001 at 25° can be determined using an Abbé refractometer or other standard apparatus

4. Nitrogen Content. 14.30±0.20%

Reagents:

a) *Standard 0.2N Titanous Chloride Solution* from Titanium Hydride (Method developed by N.M. Liszt of PicArsn is described in Vol 5 under DYNAMITE, GENERAL METHOD OF ANALYSIS APPLICABLE TO COMMERCIAL BLASTING EXPLOSIVES and also in MIL-STD-286B, Dec 1967, Method 601.1.1. This method is recommended. An older method of prepn of 0.15N $TiCl_3$ soln from 20-percent commercial Titanous Chloride soln as described in Vol 1, pp A415-R & A416-L is applicable here, except that 150ml of the 20% soln are mixed with 100ml of 38% HCl and the soln diluted to 1 liter. A detailed description of prepn is given under DYNAMITE in Vol 5

Standardization of $TiCl_3$ Solution is done by potassium dichromate as fully described under DYNAMITE or briefly under Method 601.1.1 in MIL-STD-267B, Dec 1967

Standard 0.15N Ferric Ammonium Sulfate Soln can be prepd in the same manner as described in Vol 1, p A415-R for prepn of 0.05N soln, but making it three times stronger by using 75g of sulfate for each liter of the reagent. A detailed description is given in Vol 5, under DYNAMITE and also in JAN-D-685

Standardization of Ferric Ammonium Sulfate Solution is done against std 0.2N $TiCl_3$ soln as described in Vol 5 under DYNAMITE or in JAN-D-685

Procedure for Determination of Nitrogen in Dinitroethylbenzene. Transfer an accurately weighed portion of approx 0.5g of the sample to a 250-ml volumetric flask, make up to volume with glacial AcOH and mix thoroughly. Avoid temp changes on acct of the high thermal coeff of AcOH. Displace air in the titration flask (Fig Et14) by passing a current of CO_2 from Kipp generator (Fig Et13) into the flask for 5 mins, and continue passing during the following opns. Rinse a calibrated 25-ml pipet with AcOH soln of the sample, and then transfer a 25-ml aliquot portion of the soln to the titration flask. Add an accurately measured portion of 50.00ml of the 0.2N $TiCl_3$ soln and 25ml of 15% HCl soln. Add few glass beads to the flask (to prevent bumping during boiling) and connect it to reflux condenser. Boil the mixture gently for 20 mins on a hot plate, then increase the current of CO_2 and cautiously immerse the flask with condenser attached in cold w contained in a large beaker. After the flask and contents have cooled to RT, remove the condenser and titrate the contents of the flask with the ferric ammonium sulfate soln. Near the end point (which is indicated by the fading of the color due to titanous chloride), add 5ml of 20% ammonium thiocyanate soln and continue the titration to the first red color which remains for at least 3 mins. Run a blank detn in exactly the same manner using exactly 50.00ml of the 0.2N $TiCl_3$ soln

Calculation:

$$\% \text{ Nitrogen} = \frac{0.2335(V-V_1)N}{W}$$

where:

V = ml of ferric ammonium sulfate soln required for the blank

V_1 = ml of ferric ammonium sulfate soln required to titrate the excess $TiCl_3$ after reduction of DNEtBz

N = normality of the ferric ammonium sulfate soln

W = grams of sample contd in the aliquot

5. pH Value of Water-Alcohol Extract, 5.0 to 8.0. Transfer a weighed portion of approx 30g of the sample to a 500-ml separatory funnel, and add 300ml of a mixture of 250ml distd w & 50ml of 95% ethanol. The mixt having a pH value betw 6.0 to 8.0. Shake the mixt vigorously for 1 min and allow to stand for 15 mins to allow separation into two layers. Determine the pH value of a portion of the upper water-alcohol layer, using a glass electrode

6. Benzene-Insoluble Matter, max 0.10%. Transfer a weighed portion of approx 10g of the sample to a 250-ml beaker, add 150ml benzene and stir

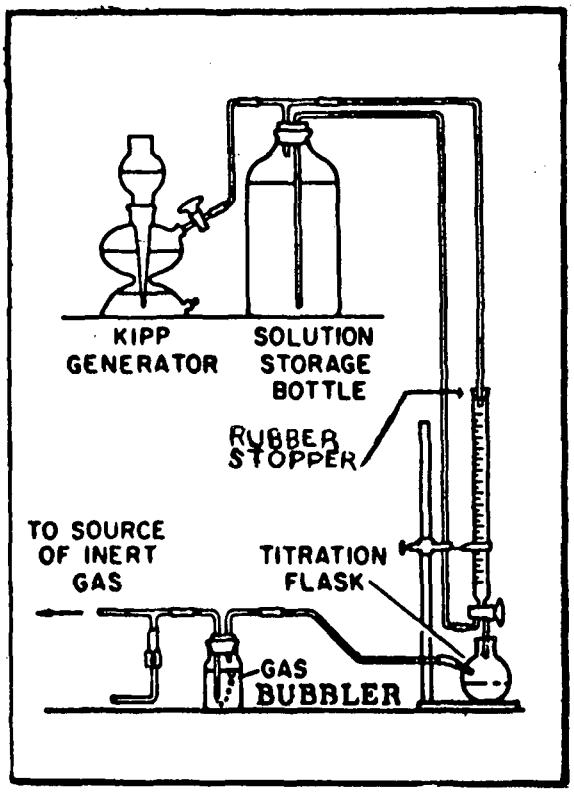


FIG Et 13 – STORAGE SYSTEM

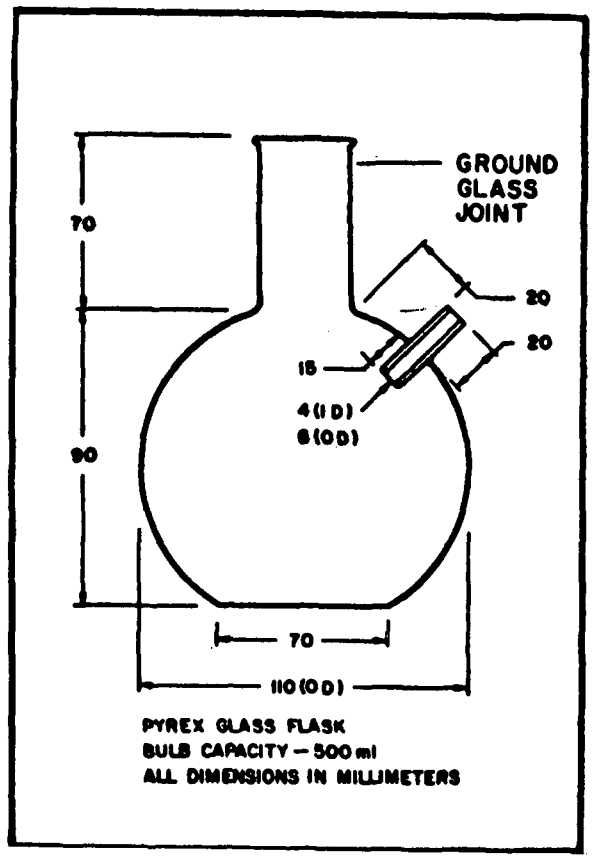


FIG Et 14 – TITRATION FLASK

the mixture until all soluble matter is dissolved. Decant the liquid and filter it thru a tared sintered glass crucible. Transfer any insoluble matter from the beaker to the crucible by means of a stream of benzene and rinse with addnl 10ml of benz. Suck air thru the crucible until only a slight odor of benz could be detected. Heat in an oven for 30 mins at 100–105°, cool in a desiccator and weigh

$$\% \text{ Insolubles} = \frac{\text{Wt of insol} \times 100}{\text{Wt of sample (10g)}} \%$$

7. *Loss on Vacuum Desiccation*, max 0.25%. Transfer a portion of approx 2g of the sample to a tared moisture detn dish, approx 2 inches in diam and reweigh the dish with the sample. Place it in a vacuum desiccator over concd sulfuric acid (95 to 97% strong) for 24 hours and then reweigh the ensemble

$$\% \text{ Loss} = \frac{\text{Loss of wt} \times 100}{\text{Wt of sample}} \%$$

1-Ethyldinitrobenzenes, $C_2H_5.C_6H_3(NO_2)_2$; Beilstein lists the following Dinitroethylbenzenes:

mw 196.16, N 14.28%, OB to CO_2 –130.05%

2,3-Dinitroethylbenzene, mp 58.5°. It may be prepd from 2,3-dinitro-4-amino-1-ethylbenzene and sodium nitrate in a mixture of alcohol and 20% oleum for 20 hours

2,5-Dinitroethylbenzene, mp 59.5°. It was prepd in a similar manner to the 2,3- derivative above

2,6-Dinitroethylbenzene, mp 57.5°. It was prepd in a similar manner to the 2,3- derivative above

3,5-Dinitroethylbenzene, mp 41°. It may be prepd by diazotization of 3,5-dinitro-4-amino-1-ethylbenzene followed by heating with alcohol

3,4-Dinitroethylbenzene, mp 124–125°. It may be prepd by treating ethylbenzene with nitric acid (d 1.38) and concd sulfuric acid at 0–10°

Ref: Beil **5**, [280] & {806}

1-Ethyl-2,4,6-trinitrobenzene or **2,4,6-Trinitro-1-ethylbenzene (TNEtB)**; listed in some papers as 2-Ethyl-1,3,5-trinitrobenzene, $C_2H_5.C_6H_2(NO_2)_3$; mw 241.16, N 17.43%, OB to CO_2 –89.6%; nearly colorless crysts, mp 37°; nearly insol in w; sl sol in alc; sol in

ether, benz and other org solvents in which TNT is sol; forms a eutectic with about 31% TNT, melting at about 2°; like TNT, it is colored red in presence of caustics. For its laboratory prepn, add slowly, with stirring, 1 part of 2,4-DNEtB to 7.25 parts of mixed acid consisting of about 22.2% nitric acid & 72.2% sulfuric acid, maintained at 75° in a stainless steel beaker, provided with an agitator and placed in a water bath. Maintain the agitation at 75° for 6 hours, disconnect the agitator, remove the beaker from water bath and allow to stand in the air until the oil (crude TNEtB) separates in the upper layer. Decant the oil into the 2nd stainless beaker, leaving the "spent acid" in the 1st beaker. Fortify the acid with nitric acid to the strength of "cycle acid", listed under prepn of 2,4-DNEtB

For purification of crude TNEtB in the 1st beaker, add hot water and agitate for a few minutes. After decanting the acidic water, agitate with a hot water Na carbonate soln, followed by 2–3 washings with hot water, under agitation. After this, the mass is grained in the following manner:

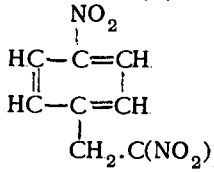
Transfer the hot oil into a flat porcelain casserole, place it on a piece of insulating material (wood, cardboard, asbestos) and stir the oil by means of a strong glass rod with a flattened end. Stir quickly with a rotary motion, occasionally pushing the crust formed at the edges back into the liquid. As soon as the oil starts to fudge, watch for lumps and break them as soon as they form with the flattened end of the above glass rod. Keep the bottom of the casserole clear of crust and continue to break the lumps. Finally the mass assumes a silky crystalline appearance and breaks into powder, which is crude TNEtB. The yield is only 75%, but it is still poorer (only 70%), if one-step nitration of EtB is used

If higher purity of the material is desired, transfer the crysts of TNEtB into a Büchner funnel (Nutsch) and wash them first with small amt of alcohol, followed by ether. After removing the rinsings by suction, dry crysts by air

TNEtB is an explosive, similar in its properties to TNT, but slightly less powerful and brisant. As it is more expensive to prep than TNT, it had no practical application in expls industry or for military purposes

Refs: 1) Beil **5**, 360 2) P. Varrato, PATR **R91**, Problem 502.6–17/41 (1930) 3) M.S. Kharash, "Preparation of Trinitroethylbenzene", OSRD Repts **285** & **295** (1941) 4) ADL, Pure-ExplCompds Part **1** (1947), p 92 5) A. Gay Lussac & H. Ficheroulle, MP **36**, 71–4 (1954) (Prepn of TNEtB) 6) L. Médard & M. Thomas, MP **36**, 97–127 (1954) & CA **50**, 3763 (1956) (Heat of Combustion and Heat of Formation) 7) G. Desseigne, MP **46–47**, 15–26 (1964-1965) (Prepn of TNEtB)

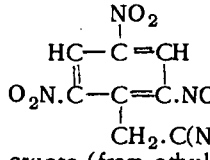
1-Nitro-4-(2,2,2-trinitroethyl)-benzene,



$CH_2.C(NO_2)_3$; mw 286.16, N 19.58%; crysts (fron alc + CCl_4), mp 135°. It may be prepd from p-nitrobenzyl bromide and silver nitroform (from silver oxide and nitroform) in ether for 50 hours. It is a powerful explosive

Refs: 1) Beil **5**, {806} 2) W.S. Reich, G.G. Rose & W. Wilson, JChemSoc, 1234–35 (1947) & CA **42**, 859 (1948)

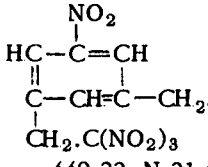
1,3,5-Trinitro-4-(2,2,2-trinitroethyl)-benzene,



$CH_2.C(NO_2)_3$; mw 376.15, N 22.34%; crysts (from ethyl nitrate + $CHCl_3$), mp 153–4°; may be prepared from 2,4,6-trinitrobenzyl bromide and silver nitroform (from silver oxide and nitroform) in ether for 18 hours. It is a powerful explosive

Refs: 1) Beil **5**, {807} 2) W.S. Reich, G.G. Rose & W. Wilson, JChemSoc, 1234–7 (1947) & CA **42**, 859 (1948)

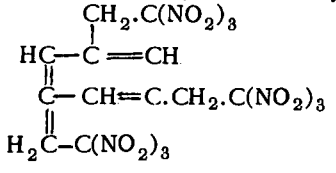
3,5-Bis(2,2,2-trinitroethyl)-1-nitrobenzene,



mw 449.22, N 21.83%; crysts (from $CHCl_3$), mp 170–171.5° (slt decompn); may be prepd from 3,5-bis(bromomethyl)-1-nitrobenzene and silver nitroform in ether for 3 days. It is a powerful explosive

Refs: 1) Beil **5**, {968} W.S. Reich, G.G. Rose & W. Wilson, JCS, 1234–7 (1947)

1,3,5-Tris(2,2,2-trinitroethyl)benzene,



mw 567.26, N 22.22%; crysts (from $CHCl_3$), mp 205–06° (decomp). It may be prepd from 1,3,5-tris-(iodomethyl) benzene and silver nitroform (from silver oxide and nitroform) in ether. It is a powerful explosive

Refs: 1) Beil **5**, {1038} 2) W.S. Reich, G.G. Rose & W. Wilson, JCS, 1234–7 (1947)

Ethylbenzene Sulfonamide and Derivatives

Ethylbenzene Sulfonamide (called Benzolsulfonsäure-äthylamid in Ger), $C_6H_5.SO_2.NH.CH_2.CH_3$; mw 185.25, N 7.6%; crysts (from alc), mp 57–58°. It can be prepd from benzenesulfochloride & ethylamine in water

Ref: Beil **11**, 40

Dinitroethylbenzene Sulfonamide, $C_8H_9N_3O_6S$, not found in the literature thru 1966

N-Trinitroethylbenzene Sulfonamide, $C_6H_5.SO_2.NH.CH_2.C(NO_2)_3$; mw 320.24, N 17.50%; crysts, mp 172°, d 1.64, ignition temp 188°, sensitivity approaches that of Comp B. It is prepd by fusing the sulfonamide with an excess of Trinitroethanol at 100°

Refs: 1) R.H. Saunders et al, HerculesPowdCo Progress Rept (Sept 1949) (Contract NOrd 9925) 2) W.F. Sager & D.V. Sickman, NAVORD Rept **483**(1952) (Res & Devel in New Chem Expls)

N-Trinitroethyl-nitrobenzene Sulfonamide, $C_8H_7N_5O_{10}S$, mw 365.25, N 19.18%. Two isomers are known:

N-Trinitroethyl-m-nitrobenzene Sulfonamide, $m\text{-}O_2N.C_6H_4.SO_2.NH.CH_2.C(NO_2)_3$; crysts, mp 149°, d 1.70, ignition temp 198°, sensitivity approaches that of TNT; prepd by fusing the sulfonamide with twice the theoretical quantity of Trinitroethanol at 90° (Refs 2 & 3). It is a powerful explosive

N-Trinitroethyl-p-nitrobenzene Sulfonamide, $p\text{-}O_2N.C_6H_4.SO_2.NH.CH_2.C(NO_2)_3$, crysts, d 1.79, ignition temp 206°, sensitivity – about the same as Comp B. It is prepd by fusing the sulfonamide with twice the theoretical quantity of Trinitroethanol at 70° (Refs 1 & 3). It is a powerful explosive

Refs: 1) R.H. Saunders et al, HerculesPowdCo ProgressRept (Oct 1948) (Contract NOrd 9925) 2) Ibid (Oct 1949) 3) W.F. Sager & D.V. Sickman, NAVORD Rept **483**(1952)

Ethylbenzoate and Derivatives

Ethylbenzoate (Called in Ref 2, Benzoic Ether), $C_6H_5.CO_2.C_2H_5$; mw 150.17; colorless aromatic liq, sp gr 1.052 at 15/15°, n_D 1.505 at RT, fr p −34.7° (−32.7°), bp 212.4° (212.9°); insol in cold w, sl sol in hot w; miscible with alc, eth, chlf & petr ether. It can be prepd by heating ethanol with benzoic acid in the presence of sulfuric acid. It is used as flavoring extract and as a solvent for many cellulose derivatives and for resins

Refs: 1) Beil **9**, 110, (62), [88] & {384} 2) CondChemDict (1961), 459-L

Ethylnitrobenzoates, $O_2N.C_6H_4.CO_2.C_2H_5$; mw 195.17, N 7.18%

Ethyl-2-nitrobenzoate, triclinic crysts, mp 30°, bp 173° at 18mm. It may be prepd from 2-nitrobenzoic acid with alcohol and K pyrosulfate and from o-nitrobenzaldehyde in alcohol

Ref: Beil **9**, 372 & (151)

Ethyl-3-nitrobenzoate, monoclinic prisms, mp 47°, bp 296°. It may be prepd by saturating an alc soln of the acid with HCl gas; from the acid salt of 3-nitrobenziminoethylether; from the 3-nitro acid and diazoethane and from the 3-nitro benz in boiling abs alc and HCl gas

Ref: Beil **9**, 378, (151), (154) & [248]

Ethyl-4-nitrobenzoate, pltlts (from alc), mp 57°. It may be prepd from the acid in alc in the presence of HCl, from the methyl ester and excess alc in the presence of $KOCH_3$

Ref: Beil **9**, 390 & [258]

Ethyl dinitrobenzoates, $(O_2N)_2.C_6H_3.CO_2.C_2H_5$, mw 240.16, N 11.66%.

Ethyl-2,3-dintrobenzoate, mp 88.4°. It may be prepd from the acid and the alcohol in the presence of hydrochloric acid

Ref: Beil **9**, 411

Ethyl-2,4-dinitrobenzoate, ndls (from alc), mp 41°. It may be prepd from the acid in alcohol in the presence of HCl gas, from the silver salt of the acid and ethyl iodide

Ref: Beil **9**, 412

Ethyl-2,5-dinitrobenzoate, crysts (from alc), mp 69.5–70°. It may be prepd from the acid and alcohol with hydrochloric acid

Ref: Beil **9**, 412

Ethyl-2,6-dinitrobenzoate, crysts (from alc), mp 74.7°. It may be prepd from the silver salt of the acid and ethyl iodide

Ref: Beil **9**, 413

Ethyl-3,4-dinitrobenzoate, mp 71°. It may be prepd from the acid and alcohol with HCl gas-from the acid chloride and ethanol

Ref: Beil **9**, 413, {1779}

Ethyl-3,5-dinitrobenzoate, ndls (from alc), sp gr 1.295 at 111°, mp 91–2°, bp – not given; insol in w; sl sol in alc. It may be prepd from the silver salt of the acid and ethyl iodide, from the acid and alc HCl; from the acid chloride in benzene; from the chloride and ether with zinc chloride

Ref: Beil **9**, 414, [280] & {1781}

2,2,2-Trinitroethylbenzoate
$C_6H_5.CO_2.CH_2C(NO_2)_3$, mw 285.17, N 14.74%, mp 72°, sensitivity about that of TNT. It may be prepd by heating benzamide and an aq mixt of nitroform, formaldehyde and nitric acid to 50°. Trinitroethanol may be used instead of nitroform and formaldehyde. It is an explosive

Refs: 1) Beil, not found 2) W.F. Sager & D.V. Sickman, NAVORD Rept **483**(1952) 3) R. Schenck, SwedP 138456(1952) & CA **48**, 2759(1954)

2,2,2-Trinitroethyl-m-nitrobenzoate,
$m\text{-}O_2N.C_6H_4.COO.CH_2.C(NO_2)_3$, mw 330.17, N 16.97%; crysts, mp 97°, sensitivity – about that of Comp B. It was prepd by nitration of Trinitroethylbenzoate. It is an explosive

Refs: 1) R.H. Saunders et al, HerculesPowdCo Progress Rept (Sept 1948) (Contract NOrd **9925**) 2) W.F. Sager & D.V. Sickman, NAVORD Rept **483**(1952) (Res & Develop in New Chem HE's)

2,2,2-Trinitroethyl-3,5-dinitrobenzoate,
$(O_2N)_2C_6H_3.COO.CH_2.C(NO_2)_3$; mw 375.17, N 18.67%; crysts, mp 138°, d 1.67, ignition temp 303°, sensitivity – about that of Comp A; prepd by treating the acid chloride & Trinitroethanol with anhyd $AlCl_3$. This compd is a good expl, but of low density

Refs: 1) M. Hill, NAVORD Rept **2245**(1951) 2) W.F. Sager & D.V. Sickman, NAVORD Rept **483**(1952)

2,2,2-Trinitroethyl-2,4,6-trinitrobenzoate,
$(O_2N)_3.C_6H_2.COO.CH_2.C(NO_2)_3$; mw 426.16, N 19.72%; crysts, mp 133–35°, d 1.80, 5 sec ignition temp 240°, hot bar ignition temp 284°, BM impact sensitivity 43cm. It was prepd from reaction of Trinitroethanol and the acid chloride in Nitrobenzene soln, or from the alcohol & acid in oleum. It is a powerful explosive

Refs: 1) Beil, not found 2) D.V. Sickman & W.F. Sager, NAVORD Rept **486**(1954)

Ethylbenzol. See Ethylbenzene

Ethyl-o-benzoylbenzoate or *Ethylester of Benzoylbenzoic Acid*(α-Oxo-diphenylmethan-carbonsäure-2-äthylester or 2-Benzoyl-benzo-säure-2-äthylester, in Ger), $C_6H_5.CO.C_6H_4.COO.C_2H_5$; mw 254.27, rhombic plates, sp gr 1.221 at 64.5°/4°, n_D 1.560 at 64.5°, mp 58°; insol in w; v sol in alc or eth. It can be prepd by treating 2-benzoylbenzoic acid with ethanol and sulfuric acid (Ref 1). Goodyear (Ref 2) proposed its use in the prepn of progressive burning double-base smokeless proplnts. For this the grains of proplnt are coated at temp below 50° with an admixture of ethyl-o-benzoylbenzoate (which is a solvent for NC), rosin, and a nonexpl, nonvolatile org compd, such as butylacetyl ricinoleate

Refs: 1) Beil **10**, 749, (356) & [517] 2) E.S. Goodyear, USP 2179330(1940) & CA **34**, 1488 (1940)

Ethyl-s-Nitro-o-benzoylbenzoate,
$C_6H_5CO.C_6H_3(NO_2).COOC_2H_5$; mw 299.29, N 4.68%, mp 86°. This ester was prepd by reacting diphenyl cadmium and 4-nitrophthalic anhydride to give the acid which was esterified

Ref: J. Tirouflet, BullSciSocBretagne Spec No **26**, 80–88(1951) & CA **47**, 8694(1953)

N-Ethyl-N-benzoylnaphthylamine-1-diazonium Hydroxide. See 4-[(N-Benzoyl-N-ethyl)-amino]-naphthalene-1-diazonium Hydroxide in Vol 2, p B90-L

Ethylbenzylamine Chlorate. See Benzylethyl-amine Chlorate under CHLORATES in Vol 2 of Encycl, p C187-L

Ethyl-bis(aminoethyl)-carbamate and Derivatives

Ethyl-N,N-bis(β-aminoethyl)-carbamate,

$H_2N.CH_2.CH_2.N(CO_2.C_2H_5).CH_2.CH_2.NH_2$.

It may be considered as a parent compd of dinitrocompd described below, although it was not used for its prepn

Ethyl-N,N-bis(β-nitraminoethyl)-carbamate,

$(O_2N)HN.CH_2.CH_2.N(CO_2.C_2H_5).CH_2.CH_2.NH(NO_2)$;

mw 265.23, N 28.41%; solid (from 95% alc), mp 104–05°; heated in a test tube, it melts and decomposes with sudden puff of fumes; when dropped on a hot plate, it burns with yel flame. It was prepd by ammonolysis of sym-Dinitrodiethylenetriurethane,

$O_2N.N(CO_2.C_2H_5).CH_2.CH_2.N(CO_2.C_2H_5).CH_2.CH_2.N(CO_2.C_2H_5).NO_2$

Refs: 1) Beil, not found 2) J.R. Johnson, OSRD **915**, 8–9, 12 & 26–27 (1942), pp 8–9

Ethyl-bis(triazomalonate) or **Diethyldiazidomalonate** (Called Diazidomalonsäure-diethylester in Ger), $(N_3)_2(COO.C_2H_5)_2$, mw 230.18, N 36.51%; pleasant smelling liquid, sp gr 1.236 at 20°; fr p ?, bp 115–115.5° at 0.81mm pressure; explodes at 180°. It can be prepd by treating diethyldichloromalonate, $Cl_2C(CO_2.C_2H_5)_2$ with NaN_3 in dil alc

Refs: 1) Beil **2**, (259) 2) M.O. Forster & R. Müller, ProcChemSoc **26**, 4(1910); JChemSoc **97**, 136(1910) & CA **4**, 1606(1910)

Ethyl Borate. See Vol 2 of Encycl, p B247-L, under Borates

Ethyl-t-butyl Peroxide, $C_2H_5–OO–C(CH_3)_3$; mw 118.17, O 27.08%; liquid, fr p –83°, bp 35° at 84mm, n_D^{20} 1.3840, d_4^{20} 0.089. It can be prepd by shaking diethylsulfate for 2 hrs with potassium t-butylperoxide

Refs: 1) Beil, not found 2) F.F. Rust et al, JACS **72**, 338(1950) & CA **45**, 1010(1951) 3) Tobolsky & Mesrobian (1954), 164

Ethylbutyramide and Derivatives

N-Ethylbutyramide,

$H_3C.CH_2–NH–CO.CH_2.CH_2.CH_3$; mw 115.17, N 12.16%; liq, bp 101–2° at 7mm, n_D 1.4372 at 20°, d 0.9028 (20°). It may be prepd from butyric acid and ethylamine by treatment with $SiCl_4$ in benzene

Refs: 1) Beil **4**, {210} 2) H. Leonormant, BullSocChemFr **1948**, p 33–46 & CA **54**, 4060 (1948) 3) Yu.K. Yur'ev et al, ZhurObshsheiKhim **29**, 2594–7 (1959)

N-(2,2,2-Trinitroethyl)-4,4,4-trinitrobutyramide,

$(NO_2)_3C.CH_2–NH–CO.CH_2.CH_2.C(NO_2)_3$; mw 385.17, N 25.45%; colorless crysts, mp 149–51°(decomp); explosive and propellant. It may be prepd by treating acrylamide with a molar equiv of HCHO to form N-methylolacrylamide on treating with 2 mols nitroform; or, acrylamide and nitroform give 4,4,4-trinitrobutyramide, which treated with formaldehyde and a second mole of nitroform gives the product

Refs: 1) Beil, not found 2) USRubberCo, Reports No 6 and Summary Report, on Contract **NOrd 10129**(1948) 3) I.J. Schaffner, USP 3038009(1962) & CA **57**, 12330(1962)

Ethylbutyrate and Derivatives

Ethyl-n-butyrate or *Ethyl Butanoate*, $C_3H_7.CO_2.C_2H_5$, mw 116.16; colorless, nontoxic, pineapple smelling liquid; sp gr 0.874 at 25°/4, fr p –100.8°(–93.3° in Ref 2), bp 121.6° (120.6° in Ref 2), n_D 1.400 at 20°; nearly insol in w; sol in alc or eth. It can be prepd by heating ethanol with butyric acid in presence of sulfuric acid, followed by distillation. It is used in flavoring extracts and in solvent mixtures for cellulose esters & ethers and for many resins

Refs: 1) Beil **2**, 270, (119), [244] & {593} 2) CondChemDict (1961), 461-L

Ethyl-4,4,4-trinitrobutyrate (EtTNBu), $(O_2N)_3C.CH_2.CH_2.CO_2.C_2H_5$, mw 251.16, N 16.73%; liq, fr p 13.4°, bp 105–06° at 2mm or 81–90° at 0.1mm; n_D 1.4537 at 25°; can be prepd by refluxing for 5 hours ethylacrylate, $CH_2{:}CH.CO_2.C_2H_5$ and nitroform, $(NO_2)_3CH$, in absolute alcohol; the potassium salt of nitroform may also be used

Refs: 1) Beil, not found 2) USRubberCo, Reports No 7 & No 8(1949) 3) K. Schimmelschmidt, GerP 852684(1952) & CA **52**, 10144 (1958) 4) J. Ville, MP **42**, 25(1960)

2,2,2-Trinitroethyl-4,4,4-trinitrobutyrate (TNEtTNBu or TNETNB), $(O_2N)_3C.CH_2.CH_2.CO_2.CH_2.C(NO_2)_3$, mw 386.15, N 21.76%; OB to CO_2 −4.1%; crysts (three polymorphs), sp gr 1.78, mp 93–4°; ignition temp 225°; v sl sol in w; sl sol in alc,; sol in eth; very resistant to hydrolysis and attack by dilute alkalies; it reacts with paraffin and waxes at elevated temps, which might interfere with its desensitization

It can be prepd either by interaction of nitroform with trinitroethylacrylate or by the esterification of trinitroethanol with trinitrobutylchloride in presence of an anhydrous metal chloride (such as $AlCl_3$) as a catalyst; US Rubber Co prepd it by treating a mixture of trinitroethanol and trinitrobutyric acid with oleum (Ref 7)

TNEtTNBu is an expl, too sensitive to impact for use as HE (16–20cm with 2kg weight); fairly stable in storage at 65°(loses 0.1–0.3% wt per year); vacuum stability at 100° – lost 0.6ml in 48 hrs

No information at our disposal for its brisance and power, but accdg to investigation conducted at Hercules Powder Co Laboratory (Ref 2), mixts TNEtTNBu with Al powder yielded about 30% more shockwave energy and 80–90% more bubble energy than did HBX-1 (RDX 40, TNT 38, Al 17, D-2 Wax 5 and Ca chloride 0.5 added)

Refs: 1) Beil, not found 2) HerculesPowderCo, Report, BUORD Contract NOrd **9925** (Nov 1948) 3) J. Rosen, NAVORD Rept **1758** (1950) (Props of TNEtTNBu) 4) ADL, Synthesis HE's, 2nd Rept, March 1951, p 33 5) M. Hill, NAVORD Lab Rept **2245**, Nov 1951 6) W.F. Sager & D.V. Sickman, NAVORD Rept **483** (1952) (Research and Development in New High Explosives) 7) USRubberCo, Quarterly Repts No **21** (1953), p 11;, No **23** (1953), pp 4 & 9 and No **24** (1953), pp 3, 8 & 9 on Contr NOrd 10129 (Prepn and props) 8) D.L. Kouba at HerculesPowderCo Laboratory, Navy Dept BuOrd Contr NOrd 11280, Task A, May 21, 1954 (Prepn and properties) 9) J. Ville, MP **42**, 23 & 25 (1960) & CA **55**, 18109 (1961) 10) R.H. Saunders, USP 2996537 (1961) & CA **55**, 26448 (1961) (Patented as an expl, the compd called 2,2,2-Trinitroethyl-γ,γ,γ-trinitrobutyrate, which is the same as 2,2,2-Trinitroethyl-4,4,4-trinitrobutyrate) 11) J.C. Conly, USP 3160654 (1964) & CA **62**, 7644 (1965) (Prepd by esterification of acid and trinitroethanol in 108% oleum; useful as proplnt and proplnt plasticizer) 12) M.E. Hill, USP 3223725 (1965) & CA **64**, 6570 (1966) 13) M.E. Hill, USP 3230247 (1966) & CA **64**, 9598 (1966) (Prepd by transesterification of methyl trinitrobutyrate with trinitroethanol in 4% oleum) 14) J.M. Rosen, J.R. Holden & H.T. Simmons, Microscope **19**, 151–6 (1971) (Crystal props of TNEtTNBu, transition temperatures, mp, heats of transition and fusion)

Trinitroethyl-trinitrobutyrate Homologs. Due to the fact that TNEtTNBu is too sensitive to impact (16–20cm with 2kg wt) for use as a booster or burster charge, attempts were made to desensitize it with substances like paraffin or waxes. It was found, however, that these substances are decompd at elevated temps by TNEtTNBu. Since it was felt that expls in which one or two NO_2 groups are replaced by CH_2 might prove (being less sensitive than the original compd) suitable for the purposes indicated above. For this reason several homologs were prepd at Laboratories of Hercules Powder Co and of Naval Ord at White Oak, Silver Spring, Md. The first homologs prepd for the US Navy were Dinitropropyl-trinitrobutyrate and 2,2,2-Trinitroethyl-4,4-dinitropentanoate. As the characteristics of these compds seemed to be favorable (notwithstanding their lack of oxygen), several other homologs were prepd for the Navy. Prepn and props of these homologs were described in confidential reports (declassified by now), listed below as refs:

Refs: 1) W.F. Sager & D.V. Sickman, NavOrd Rept **483**, June 18, 1952 (Research and development in new HE's) 2) D. Jensen, NavOrd Rept **2498** (1952) (Prepn of 2,2,2-Trinitroethyl-4,4-dinitropentanoate) 3) D.L. Kouba & H.D. McNeil, HerculesPowderCo Summary Rept, 48 **2092**, May 21, 1954 4) D.L. Kouba, HerculesPowderCo, ProgressRept on High Explosives, Navy Contract NOrd **11280**, Task A, Aug 18, 1955

Ethylcarbamate or Urethane. See Carbamic Acid Ethyl Ester in Vol 2, pp C40-R & C41-L

The following derivatives are not described there:

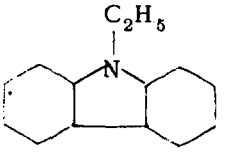
Ethylcarbamate Azide (Äthylcarbamidsäure-azid in Ger), $C_2H_5.NH.CO.N_3$; mw 114.11, N 49.10%; prisms, mp 10–14°, bp 90° at 28mm. It may be

prepd from ammonia and ethyl isocyanate in ether
Refs: 1) Beil **4**, (354) 2) N. Oliveri-Mandalà, GazzChimItal **43**, 309(1913)
Nitroethylcarbamate, $O_2N.NH.CO_2.C_2H_5$; mw 134.09, N 20.89%; tablets (from eth + petr eth), mp 64°; sol in water, alc, eth. It may be prepd from ethyl nitrate and urethane in concd sulfuric acid
Ref: Beil **3**, 125

Trinitroethylcarbamate, $H_2N.COO.CH_2.C(NO_2)_3$; mw 224.09, N 25.00%; crysts, mp 94°, d 1.79, ignition temp 214°, sensitivity – about the same as Pentolite. This compd is relatively stable at 90°, but much less stable at 100° in liq state. It is prepd from carbamyl chloride & Trinitroethanol
Refs: 1) D.L. Kouba & R.H. Saunders, HerculesPowdCo Progress Rept (May 1950) (Contract NOrd **9925**) 2) W.F. Sager & D.V. Sickman, NAVORD Rept **483**(1952)

2,2,2-Trinitroethylcarbamic Acid, 2,2,2-trinitroethylester,
$(NO_2)_3C.CH_2.HN.CO_2.CH_2.C(NO_2)_3$; mw 387.14, N 25.30%; mp 192°. It may be prepd from trinitroethanol, nitric acid and urea in 92–5% yield in presence of 10% water; 75% yield with 40% water; $HC(NO_2)_3$ + HCHO may be substd for $(NO_2)_3C.CH_2OH$. It is an explosive
Refs: 1) Beil, not found 2) R. Schenck, SwedP 138456(1952) & CA **48**, 2759(1954)

Ethylcarbazole and Derivatives
Ethylcarbazole,



mw 195.25, lflts (from eth), mp 67–70°, bp 175° at 5mm; insol in w; sol in eth & hot alc. It can be prepd by action of ethyl chloride on K carbazole. Since its props are similar to Carbazole, described in Vol 2, p C45, it might be suitable for use as a stabilizer
Refs: 1) Beil **20**, 436, (164) & [282] 2) Cond-ChemDict(1961), 462-R

9-Ethylnitrocarbazoles, $O_2N.C_{12}H_8.C_2H_5$; mw 241.26, N 11.61%
9-Ethyl-2-nitrocarbazole, crysts, mp 148.5–149°. It may be prepd by treating 2-nitrocarbazole in acetone with aq KOH (66%) and followed by ethyl chloride
Ref: E. Sawicki, JACS **75**, 4106–7(1953) & CA **49**, 8243(1955)
9-Ethyl-3-nitrocarbazole, yel crysts (from alc), mp 128°. It may be prepd from ethyl carbazole, $NaNO_2$ and HCl in boiling benz or with 43% HNO_3 at 10–20°; from 3-nitrocarbazole and Ethyl bromide or iodide followed by diethylsulfate in alkali; from ethylcarbazole and tetranitromethane in acet ac and CH_2Cl_2, irradiation with ultraviolet light cuts reaction time from 4 wks to 90 mins
Refs: 1) Beil **20**, 440, (168) & [288] 2) D.H. Iles & A. Ledwith, JCS(1969), 364–5 & CA **70**, 114937(1969)

9-Ethyl-1,3,6,8-tetranitrocarbazole,
$(O_2N)_4.C_{12}H_4N.C_2H_5$; mw 375.26, N 18.67%; dimorphic yel ndls, mp 203°, 216°; sol in acetone. It may be prepd from 3,3′,5,5′-tetranitro-2,2′-dimethoxybiphenyl in alcohol and ethylamine by heating 5 hrs at 100°. It is an explosive
Refs: 1) Beil, not found 2) I.J. van Alphen, RecTranChim **51**, 179–84(1932) & CA **26**, 1273(1932)

Ethylcarbonate and Derivatives
Ethyl- or Diethylcarbonate, $(C_2H_5O)_2CO$; mw 118.13, colorless liq with mild odor, sp gr 0.975 at 20°/4, fr p –43°, bp 126.8°; fl p 115°F (open cup); combustible but not inflammable; insol in w; miscible with alcs, ketones, esters, aromatic hydrocarbons and some aliphatic solvents. It cannot be prepd by the esterification process because carbonic acid is not reactive with alc. The successive steps in its manuf are: a) reacting Cl and CO to produce phosgene ($COCl_2$); b) reacting phosgene with ethanol to make ethylchlorocarbonate ($ClCO_2C_2H_5$); and c) reacting ethylchlorocarbonate with anhydrous ethanol to produce diethylcarbonate. After these steps, the crude product is neutralized and redistilled
It is used as solvent for NC, cellulose ethers, resins, radio tube cathode fixing lacquers and in org syntheses

Refs: 1) Beil **3**, 5, (4), [4] & {5} 2) Cond-ChemDict (1961), 374-R (Diethylcarbonate)

Hexanitroethylcarbonate, $[(O_2N)_3C.CH_2O]_2CO$; mw 388.13, N 21.65%; crysts, mp 115°, d 1.88, sensitivity approx that of Pentolite. It is prepd by reaction of phosgene with Trinitroethanol in the presence of $AlCl_3$. This expl has a high oxygen content (62%), high crystal density and excellent stability
Ref: D.V. Sickman & W.F. Sager, NAVORD Report **486** (1954)

Ethylcelluloses (EtCell) are esters of cellulose in which one or more (up to three) hydroxyl groups are replaced by ethoxyl groups ($-OC_2H_5$). The monoethylcellulose, $C_6H_7O_2(OH)_2(OC_2H_5)$, contains 23.68% of (OC_2H_5), the di-, $C_6H_7O_2(OH)(OC_2H_5)_2$, contains 41.28% (mp about 150°) and the tri-, $C_6H_7O_2(OC_2H_5)_3$, contains 54.88% of (OC_2H_5) (mp 240–245°)

The completely substituted product is a white, granular, solid with mw 246.30 and mp 240–45°. Its method of prepn was described by Hess & Müller (Ref 1). The triethylcellulose is insol in w and practically insol in methanol, ethanol & acetone, but sol in chlf, pyridine, ethylene dichloride, ethyl acetate, carbon tetrachloride and many other organic solvents. This product has, however, no military or commercial application because it lacks strength & flexibility, is not thermoplastic and shows only very limited compatibility

A product which contains about 2.5 ethoxy groups (47–48%) is thermoplastic and is suitable for commercial and military uses; its sp gr is 1.07–1.18, n_D 1.47 and softening point 240–45°. It can be prepd in a laboratory, by slowly adding (with stirring) to the pulped filter paper (suspended in 44% aq NaOH soln maintained at 100°) an excess of ethyl chloride (usually 6 moles per one mole of cellulose) and allowing to stand for a while
Note: In order to obviate to some extent the side reactions of hydrolysis, the procedure is conducted in the presence of large amount of NaCl

The resulting slurry is filtered thru a Gooch and the pulp sucked fairly dry. If analysis will show that the ethoxyl content is lower than desired, the treatment with ethyl chloride can be repeated (Ref 3). Technical methods of prepn are described by Dorée (Ref 4)

R.F. Warren, Edit, gave in Ref 5 a rather detailed description of manuf of military grade EtCell as was conducted after WWII in the USA. The description, which is accompanied by schematic diagram, is not included here since it is described in the literature

Five types of commercial EtCell were manufd during WWII by the Hercules Powder Co (Ref 2), of which the **N-Type**, having 46.8 to 48.5% ethoxyl content, had sp gr 1.14, mp 200–210° and the same requirements as were listed in USArmySpecification No 97–54–176, now replaced by Spec MIL-E-10853B. The product is more resistant to alkali solns than to the acids. Its flammability is of as low an order as any other cellulosic derivatives (Ref 2)

Uses of EtCell include: films, adhesive, flexible lacquers, varnishes, plastics, electrical insulators, moulding powders, jelled expls such as used in Bangalore Torpedo (See Vol 2, pp B16 ff), inhibitor tape for rockets and proximity fuzes (Refs 8 & 10)

Most renowned of EtCell uses is the proximity fuze developed during WWII. Out of many plastic materials tested, EtCell was chosen because of its toughness, close-tolerance moulding and heat resistance. All this in spite of the fact that it is a thermoplastic material. (See also Refs 9, 11 & 12)
Refs: 1) K. Hess & A. Müller, Ann **455**, 209 (1927) 2) HerculesPowderCo Pamphlet, entitled "Ethyl Cellulose", Wilmington, Del 3) J.T. Marsh & F.C. Wood, "An Introduction to the Chemistry of Cellulose", Chapman Hall, London (1945), 368–88 4) C. Dorée, "The Methods of Cellulose Chemistry", Chapman Hall, London (1947), 318–22 5) J. Cyrot, MémServChimÉtat, **33**, 159 (1947) (Étude de l'éthylation de la cellulose sous vide) 6) A. Tribot & A. Maraudon, Ibid **33**, 197 (1947) (Éthylation de la cellulose) 7) J. Chédin & A. Tribot, Ibid **33**, 169 (1947) & **34**, 195 (1948) (Méthode d'obtention d'éthylcellulose) 8) R.F. Warren, Edit, "Ethyl Cellulose", ChemEngrg, March 1951, p 279 9) J.M. Caraher, "Development of an Ethylcellulose Inhibitor Formulation", NAVORD Rept **4977** NOTS 1304) (Sept 1957) 10) Cond-ChemDict (1961), 462-L 11) Kirk & Othmer,

2nd edit 4(1964), 638–92 12) E. Ott, "Cellulose Derivatives", Interscience, NY, Vol 5, Part 5(1971), 785 13) CondChemDict (1971), 360–61

Ethyl Cellulose. Requirements and Tests of the US Armed Forces Specification MIL-E-10853B, Dec 1958

This spec covers one grade of EtCell but two classes: Class 1 is of high viscosity, while Class 2 is of low viscosity. Class 1 material shall fuse without charring and be capable of being drawn into a thread at a temperature not lower than 145°C, when tested as specified in opn 8

The EtCell shall be white, porous granules of freshly prepd stock, manufd from purified cellulose

Each lot shall consist of EtCell produced by one manufacturer in not more than 24 consecutive hours under essentially the same manufg conditions and with no change in materials, provided the operation is continuous. In the event that the process is a batch operation, each batch shall constitute a lot

A batch is defined as that quantity of material which has been manufd by some unit chem process and subjected to some physical mixing opn intended to make the final product substantially uniform

The EtCell covered by this Spec is intended for use in the manuf of certain munition components (which are not named in the Spec but were named above)

Sampling for examination shall be conducted in accordance with Standard MIL-STD-105. A sample shall be taken from each lot, the size to be calcd on the basis of one-tenth of the square root of the number of containers in the lot, raised to the next highest whole number. If there are fewer than 3 containers in a lot, each container shall be sampled. In all other cases, no fewer than 3 containers shall be selected. From each container in the sample a representative one pound specimen shall be taken and placed in separate, clean, dry containers which are labeled to identify the lot and container represented. Equal portions of each specimen selected, as described above, shall be thoroughly mixed to form a composite specimen, and this shall be subjected to the tests described below

Specification Requirements and Tests

1. Ash Content – 0.40% max for Class 1 and 0.10% for Class 2

Transfer approx 5g of the specimen, weighed to the nearest mg, to a tared porcelain crucible and ignite for 4 hrs in a muffle furnace maintained at 900±25°C, cool in a desiccator and weigh to the nearest mg

$$\% \text{ Ash} = 100A/W$$

where:

A = wt of residue after ignition of specimen, in g

W = wt of specimen in g

2. Ethoxy Content – 46.8 to 48.5% for Class 1 and 47.1 to 48.1 for Class 2

Prepare on the day of test a soln of bromine-Na acetate–acetic acid soln by adding 10ml of bromine to 145ml of 10% soln of anhydrous Na acetate in glacial AcOH

Procedure: Assemble the apparatus (Fig Et 15) for the ethoxy detn. Grind approx 1 g of the specimen to pass thru 149 micron (No 100) sieve conforming to Spec RR-S-366 and dry it at 100°±5° for 2 hrs. Fill the trap T by pouring a small amt of aq suspension of red phosphorus thru the cup C above. Follow with a water rinse, using sufficient liq to make the trap about half full. Weigh to the nearest 0.1mg appr 50mg of dried sample placed in a tared gelatin capsule. Transfer the capsule to the boiling flask, add a few glass beads and 6ml of constant boiling 57% hydriodic acid, but do not start heating the flask yet. Place 10ml of bromine-Na acetate-acetic acid soln in the 1st receiver and 6ml in the 2nd one. Place the 2nd receiver with contents under the side arm of the 1st receiver until the side arm is approx 0.5 inch from the bottom of the 2nd receiver. Purge the ethyl iodide formed in the boiling flask into the contents of the 1st receiver by bubbling a slow stream of CO_2 first thru a scrubber tower contg 100ml of concd sulfuric acid and then thru the side arm of the boiling flask at the rate of approx 2 bubbles per sec. Place the flask in an appropriate bath and maintain at 150°C for 40 mins, while collecting the distillate in the 1st and 2nd receivers. Remove the source of heat, stop bubbling CO_2 and wash the contents of both receivers into a 500-ml flask contg 20ml of a soln contg 250g of Na acetate per 1 liter of distd w. Dilute to 125ml e ith water, add

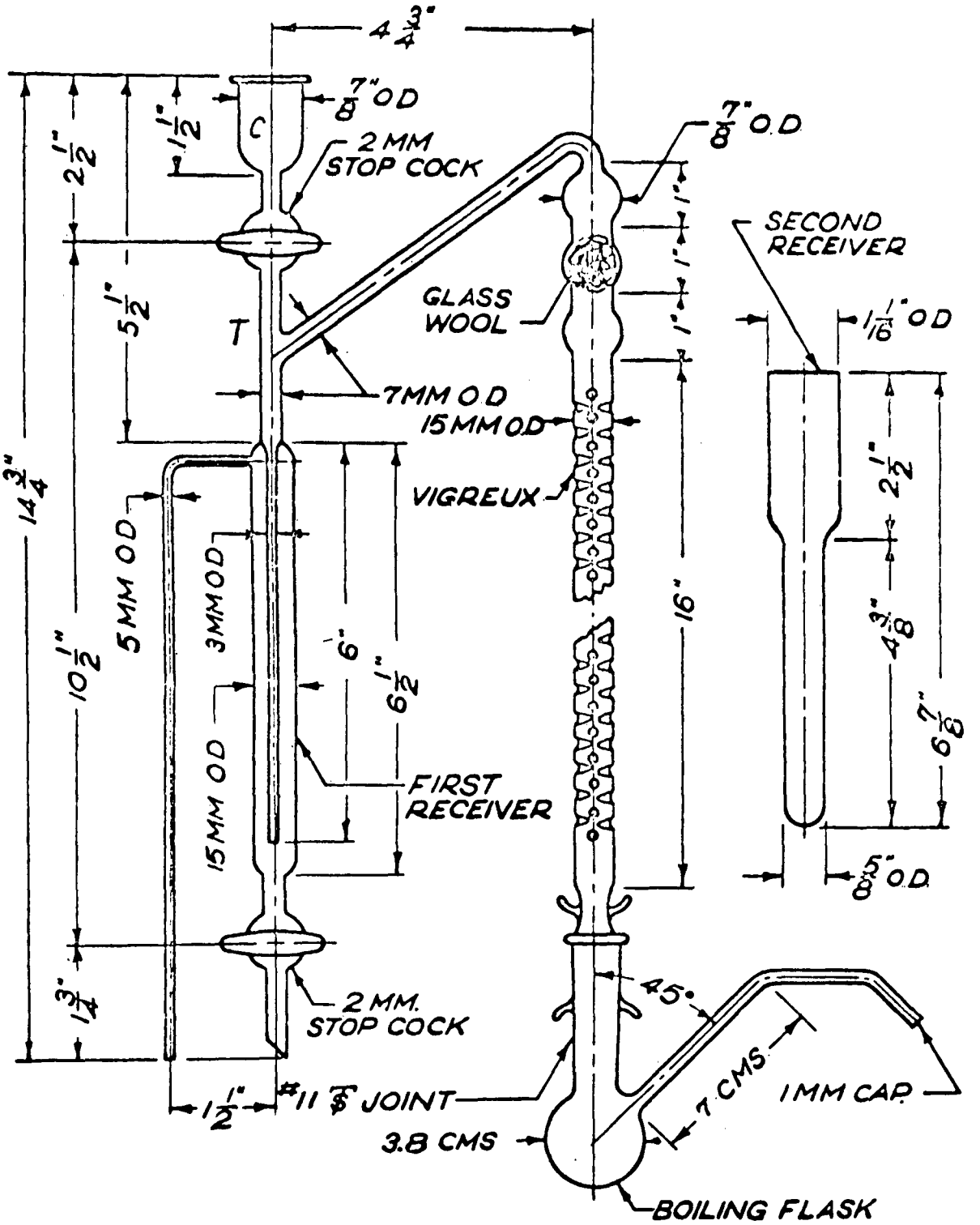


FIG Et15 – ETHOXY APPARATUS

90% soln of formic acid dropwise with swirling until the liquid becomes colorless, and then add 3 drops more. This usually requires a total of 12 to 15 drops. Allow 3 mins for the desired reaction to be completed and then add 25ml of 10% sulfuric acid and 3g of KI. Swirl the mixture and, after allowing to stand for 3 mins (while protected from light), titrate the liberated iodine with 0.1N Na thiosulfate soln using starch as an indicator

% Ethoxy = 0.751 AN/W

where:

A = ml of Na thiosulfate soln used for specimen

N = its normality

W = wt of specimen, corrected for ash content

3. *Moisture Content* – maximum 2.0% for Class 1 and 2.2% for Class 2

Weigh to the nearest 0.1mg approx 5g of the specimen in a tared short, wide-mouth weighing bottle, equipped with a glass stopper. Heat without stopper for 1 hour at 100 to 105°, close loosely, cool in a desiccator and weigh. Repeat the heating until constant wt is obtd. Calculate the loss in wt as % of moisture in the specimen

4. *Acidity* – none in Class 1 and in Class 2

Boil 10.00g of the specimen with 150ml freshly distd water for 5 mins, allow to settle, and cool. Titrate with weak std KOH (or NaOH) soln using red as an indicator

5. *Chloride Content as NaCl* – maximum 0.02% for Class 2

Weigh 10g of the specimen to the nearest 0.01g and transfer to a 500-ml Erlenmeyer flask. Add 150ml of distd w and 40ml of 1:3 soln of HNO_3. Heat the flask to boiling its contents and continue to boil for 2 minutes. Filter thru a Gooch crucible into a flask and rinse the filter and the flask with four 5-ml portions of water, into the same flask. Add 5ml of approx 0.1N Ag nitrate soln and 3ml of pure Nitrobenzene, followed by 1 ml of ferric alum soln. Shake vigorously to coagulate the ppt and titrate the excess of Ag nitrate with std 0.1N K thiocyanate soln until a reddish-brown color appears that does not fade in 5 minutes

% Chloride as NaCl = [5.845(AB–CD)]/W

where:

A = ml Ag nitrate soln used

B = its normality

C = ml of K thiocyanate soln used in titration

D = its normality

W = wt of specimen used, in g

6. *Viscosity at 25°C*(5% EtCell in 80:20–toluene-ethanol soln) centipoises – 10 ± 1 for Class 1 and 7 ± 1 for Class 2

Dissolve 5.00g of the specimen in 76g of toluene and 19g of ethanol conforming to Spec O–E–760. Determine the viscosity at 25° according to Method 305 of Federal Test Method Standard No 791, except that the results in centistokes should be converted to centipoises as follows:

Viscosity in centipoises = dv

where:

d = density of EtCell soln

v = kinematic viscosity in centistokes

Note: Density can be detd by any suitable method accurate to three significant figures, such as pycnometer method described in Vol 3 of Encycl, p D69

7. *Granulation* – maximum 1.0% for Class 2 only, retained on a 2000 micron (No 10) sieve

Weigh approx 100g of the specimen to the nearest 0.1g and place on a No 10 sieve conforming to Spec RR–S–356 and equipped with a cover and a bottom pan. Shake the sieve for 5 mins in a mechanical shaker geared to produce 300 ± 15 gyrations and 150 ± 10 taps of the striker per minute. Weigh the portion retained on the sieve and calculate the % retained

8. *Thermoplastic Point.* Class 1 material shall fuse without charring and be capable of being drawn into a thread at a temp not lower than 145°C

Procedure: Fill a ¾x4-inch test tube to a depth of 1 inch with the specimen ground to pass a No 16 mesh screen. Submerge the tube to a depth of 2 inches in an oil bath previously heated to 120°, place a thermometer in the EtCell and increase the bath temp at the rate of 2°C per min, while stirring the specimen by means of thermometer. At a temp approx 25° below fusion point the particles soften and tend to cohere, making it difficult to stir. Continue heating and working the mass until it fuses and becomes plastic. Read the "thermoplastic point" as the temp at which the plastic mass can be repeatedly drawn into thread by pulling the thermometer from the mass. Duplicate dets should check within 3°C

Ethyl Centralite or **sym-Diethyldiphenylurea.** See Centralite 1 or Carbamite in Vol 2 of Encycl, pp C127 to C129. Analytical procedures including tests prescribed by US Joint Army-Navy Specification JAN-E-255 are described on pp C129 to C134

Ethyl Chlorate. See Ethyl Chlorite below

Ethyl Chloride. See Chloroethane in Vol 3, p C254-L and CondChemDict (1961), p 462-L

Ethyl Chlorite, $C_2H_5.ClO_2$. The only ref to this compd or the corresponding chlorate reported an attempt to prepare ethyl perchlorite from ethyl iodide and silver chlorite at 15°. No reaction was noticeable until the solution was allowed to warm a little, after which some AgI precipitated. The solution exploded a short while later
Ref: G.A. Levy, GazzChimItal **53**, 40–2(1923) & CA **17**, 2263(1923)

Ethylchloroacetate, $Cl.CH_2.CO_2.C_2H_5$; mw 122.55; colorless pungent liq, sp gr 1.159 at 20°/4, fr p –26°, bp 144°; flash p 54°C; insol in w; sol in alc, eth or benz. It can be prepd by the action of chloroacetyl chloride on ethanol or by treating chloroacetic acid with ethanol and sulfuric acid. It is used as solvent and as military poison gas. It was not described under Chemical Warfare Agents in Vol 2 of Encycl
Refs: 1) Beil **2**, 197, (88), [191] & {440}
2) CondChemDict (1961), p 462-R

Ethyl Chlorocarbonate or **Ethylchloroformate,** $Cl.CO_2.C_2H_5$; mw 108.53, colorless, toxic, flammable, tear-producing liq; sp gr 1.138 at 20°/4, fr p –80.6°, bp 94–5°, flash p 61°F (approx); insol in w, but decompg; sol in alc, eth, benz & chlf. It can be prepd by reacting CO with gaseous chlorine, producing phosgene ($COCl_2$) which is then reacted with anhydrous ethanol, giving ethyl chlorocarbonate and splitting off HCl. It is used in organic synthesis as an intermediate in production of diethylcarbonate
Refs: 1) Beil **3**, 10, (5), [10] & {24}
2) CondChemDict (1961), 462-R

Ethylchloroformate. See Ethylchlorocarbonate

Ethylchlorosulfonate. French Chemical Warfare Agent Villantite, listed in Vol 2, p C169 and in CondChemDict (1961), 463-R

Ethylcresol and Derivatives
5-Ethyl-m-cresol, $H_5C_2.C_6H_3.(CH_3).OH$; mw 136.19; prisms (from benz), mp 55°, bp 232.5°, 130° at 20mm. It may be prepared from m- or p-cresol and ether in the presence of $AlCl_3$
Refs: 1) Beil **6**, {1822} 2) E.C. Horning et al, JACS **71**, 169(1949)

5-Ethyl-2,4,6-trinitro-m-cresol,
$H_5C_2.(CH_3)C_6(NO_2)_3.OH$; mw 271.18, N 15.50%, OB –56.0%, crysts (from petr eth), mp 67.0°. It may be prepd by dissolving 3-methyl-5-ethylphenol in sulfuric acid and adding nitric acid, drowning on ice, and recrystallizing
Refs: 1) Beil, not found 2) C. E. Moore & R. Peck, JOrgChem **20**, 673–9(1955) & CA **50**, 2290(1956)

Ethyl Cyanide or **Propionic Nitrile,** $C_2H_5.CN$; mw 55.08; colorless, toxic liq with ethereal odor; sp gr 0.7829 at 20°/20, n_D 1.3664, fr p –91.9°, bp 97.2°, flash p 61°F (open cup); insol in w; sol in alc & eth. It can be prepd by heating Ba ethyl-sulfate with KCN, followed by distillation. It is used as solvent, dielectric fluid and as an intermediate
Refs: 1) Beil **2**, 245, (109), [225] & {547}
2) CondChemDict (1961), 463-L

Ethylcyanoacetate or **Cyanacetic Ester,** $NC.CH_2.CO_2.C_2H_5$; mw 113.11, colorless liq, sp gr 1.062 at 20°/4, fr p –22.5°, bp 208° at 753mm; sl sol in w & in alkaline solns; sol in alc & meth. It can be prepd by esterification of cyanoacetic acid with ethanol or by reaction of an alkali cyanide with chloroacetic ethyl ester. It is used in org synthesis

Note: Accdg to Kay-Fries Catalog (fine chemicals), it contains the highly reactive "cyano-activated" methylene group
Refs: 1) Beil **2**, 585, (254), [531] & {1628} 2) CondChemDict (1961), 463-R

Ethylcyanoformate. A Chemical Warfare Agent listed in Vol 2, p C169-R as item t)

Ethylcyanomethylamine and Derivatives
N-Ethyl-N-cyanomethylamine (Also called Ethylmethylcyanamide), $C_2H_5(CH_3)NC{:}N$; mw 84.12, N 33.30%. It may be prepd from ethyldimethylamine and BrCN which give $Et(Me)_2N^+C{:}N\ Br^-$. Heating gives $Et(Me)NC{:}N$ and CH_3Br
Refs: 1) Beil, not found 2) F. Möller, Methoden der Organisch Chemie (Houben,Weyl), Vol **11/1**, p 982(1957) 3) D. Marten and W.M. Brause, JPractChem **1971**, 312(5), pp 812–15 & CA **74**, 132585(1971)

N-Trinitroethyl-N-cyanomethyl Nitramine,
$(O_2N)_3C.CH_2.N(CH_2.CN).NO_2$; mw 265.12, N 31.63%; crysts, mp 81–82°, d 1.75g/cc, ignition temp 194°, impact sensitivity 11cm with 2½kg wt, thermal stability 16.8cc/g at 90°. Its method of prepn is not reported in Ref 2. Thi s compd was not of interest due to it s poor thermal stability
Refs: 1) Beil, not found 2) D.L. Kouba et al, HerculesPowdCo Progress Rept, May 1952 (Contract NOrd-11280, Task A)

1-Ethylcyclohexyl Hydroperoxide-p-Nitrobenzoate,
$(CH_2)_5.C(C_2H_5)O_2.CO.C_6H_4.NO_2$; mw 293.33, N 4.77%; mp 68–9°; expl in a flame. Criegee & Dietrich (Ref 2) treated 1-ethyl cyclohexanol with 83% hydrogen peroxide and 0.2cm concd sulfuric acid for 14 hrs at 0° and purified the Na salt. The peroxide and p-nitrobenzoyl chloride in abs pyridine gave the nitrobenzoate as yellow plates from ether or methanol
Refs: 1) Beil, not found 2) R. Criegee & H. Dietrich, Ann **560**, 139(1948) & CA **43**, 6189(1949)

Ethyldiaminomethane and Derivatives
N-Ethyl-diaminomethane or *N-Ethyl-methylene-diamine*, $CH_3.CH_2.NH.CH_2.NH_2$, called N-Ethylmethanediamine in CA and $CH_3.CH(NH_2).CH_2.NH_2$, called *Propylene-1,2-diamine*, mw 74.12, N 37.79%
Refs: 1) Beil, not found 2) CA, not found

NOTE: The nitrated compd, $CH_3.CH(NH.NO_2).CH_2(NH.NO_2)$, listed below, is also found in Vol **5**, p D1142-R under 1,2-Dinitraminopropane or Propylenedinitramine

N-Ethyl-N,N'-dinitroaminomethane or **N-Ethyl-methylenedinitramine,**
$CH_3.CH_2.N(NO_2).CH_2.NH(NO_2)$; mw 164.13, N 34.14%, OB to CO_2 –58.5%. This compd is an expl having 118% the power of TNT by ballistic mortar test. It is listed by Blatt as *Trimethylene-1,3-dinitramine*

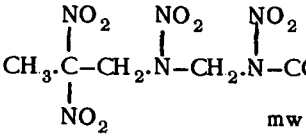
Blatt (Ref 2) also lists *Trimethylene-1,2-dinitramine* or *Propylene-1,2-dinitramine (PDNA)* with the formula, $CH_3.CH(NH.NO_2).CH_2.(NH.NO_2)$, crysts, mp 108.5–110°; solubilities – not given. It can be prepd by treating $CH_3.CH(NH_2).CH_2.NH_2$ with $ClCOOC_2H_5$ (ethyl chlorocarbonate) to obtain $CH_3.CH(NH.CO_2C_2H_5).CH_2.NH.CO_2C_2H_5$, followed by nitration & hydrolysis

It is an expl compd, possessing the following properties:
Hygroscopicity at 25°: gained 0.76% at 90% RH and 2.42% at 100% RH; *Ignition Temperature* 355°; *Impact Sensitivity* with 2kg wt using Bruceton No 3 Machine – 50% positive at 63cm drop; *International Test at 75°* – lost 2%; *Power by Ballistic Mortar* – 116% TNT; *Power by Trauzl Test* – 123% TNT; *Stability at 100°* – not acid in 300 mins; *Vacuum Stability at 100°* – lost >12cc/5g in 24 hours (Refs 2 & 3)
Refs: 1) Beil, not found 2) Blatt, OSRD **2014** (1944), under Trimethylene-1,2-dinitramine, taken from NDRC, Div 8, Interim Rept **PT-9**, Apr 15 to May 15, 1943, p 29 3) ADL, Pure-ExplCompds, Part **1**(1947), p 34

Ethyldiazaheptanoate and Derivatives
Ethyldiazaheptanoate, $CH_3.CH_2.CH_2.NH.CH_2.NH.CO_2.CH_2.CH_3$; may be considered as the parent compd of its nitrated derivs, although not used to prep them

2,2,2-Trinitroethyl-4,6,6-trinitro-2,4-diazaheptanoate, $CH_3.C(NO_2)_2.CH_2.N(NO_2).CH_2.NH.COO.CH_2.C(NO_2)_3$; mw 430.21, N 26.05%; viscous brown oil. It was prepd by converting 3,5,5-trinitro-3-azahexanoyl chloride to the corresponding isocyanate and treating with Trinitroethanol in chloroform (Ref 2). Its expl props were not detd

2,2,2-Trinitroethyl-2,4,6,6-tetranitro-2,4-diazaheptanoate,



mw 475.21, N 26.53%; wh crysts (from ethylene dichloride); mp 140–41°; impact sensitivity – 25cm with 2kg wt; power by Pb block 167% (calcd) & by ballistic mortar 144% TNT (calcd)

This compd was prepd by adding dropwise the above trinitro deriv in acetic anhydride to 100% nitric acid cooled to 5°. The soln was stirred for 20 mins at 5° and poured onto ice (Ref 2). Heat of combustion 1806cal/g (Ref 2)

Other props reported are crystal d 1.79, hot bar ignition temp 219°, sensitivity – same as Pentolite (Ref 3)

Refs: 1) Beil, not found 2) L.T. Carleton & M.B. Frankel, AerojetGeneralCorp Quarterly Rept **711** (June 1953), 10–12 3) D.V. Sickman & W.F. Sager, NAVORD Rept **486** (1954)

Ethyl Diazide. See Ethane Diazide in this Vol

Ethyldiazidomalonate. See Ethyl-bis(triazomalonate) in this Vol

Ethyldiazoacetate or **Diazoethylacetate,** $N_2{:}CH.CO_2.C_2H_5$; mw 114.10, yel oil (which is easily converted to solid modification), sp gr 1.085 at 18°/4, fr p –22°, bp 140–41° at 720mm; sl sol in w; sol in alc & eth. It can explode during vacuum distillation; may be prepd by diazotization of ethyl glycinate with HCl and $NaNO_2$ or by reaction of ethyl glycinate.HCl, $NaNO_2$ and 10% H_2SO_4 in ether or chlf

Refs: 1) Beil **25**, 110; **3**, (211), [390] & {1140} 2) J.A.S. Hammond, USP 2691649 (1954) & CA **49**, 11690 (1955); USP 2691650 (1954) & CA **49**, 11691 (1955) 3) P. Yates, B. Shapiro, N. Yoda & J. Fugger, JACS **79**, 5756–60 (1957)

Ethyldiazosuccinate or **Diazoethylsuccinate,** $C_2H_5.O_2C.CH_2.C(N_2).CO_2.C_2H_5$; mw 200.19, N 13.99%; yel liq, crysts (from a cold mixt of eth & petr eth), bp 77–78° at 0.1mm press; sp gr 1.139 at 20°, n_D 1.4620 at 20°

An attempt to prepare ethyl diazosuccinamate from ethyl diazosuccinate and liq NH_3 resulted in a violent expln (Ref 2). Ethyl diazosuccinate is prepd by diazotization of diethylaminosuccinate (Ref 1)

Refs: 1) Beil **25**, 159; **3**, (274), [479] & {1364} 2) A. Darapsky, Ber **43**, 1102 (1910) & CA **4**, 2466 (1910)

Ethyldichloramine. See N,N-Dichloroethylamine in Vol **5**, p D1210-R

Ethyldichloroarsine. See Chemical Warfare Agent **ED** in Vol 2, p C167-R

Ethyldichlorosulfide or **Dichloroethyl Sulfide.** See Chemical Warfare Agent **H** (or **HS**), known in the US as *Mustard Gas*, in France as *Ypérite* and in Germany as *Gelbkreuz* or *Lost*

Ethyl Diethanolamine and Derivatives

Ethyl Diethanolamine (EDEtA), $C_2H_5N(CH_2CH_2OH)_2$; mw 133.19, N 10.52%, colorless liq with amine odor, sp gr 1.015 at 20°, n_D 1.4663 at 20°, bp 251–52°; flash p 255°F, sol in water & alc; prepd by adding ethylene oxide to a cold solution of ethylamine. It is used as solvent and in detergents

Refs: 1) Beil **4**, 284 2) L. Knorr & W. Schmidt, Ber **31**, 1074 (1898) 3) CondChemDict (1961), 464-L

Ethyl Diethanolamine, US Military Requirements and Tests, as described in Specification MIL-E-10660B (CmlC), 27 Apr 1959. Accdg to Notice 1, 3 Feb 1972, the material covered by the spec was used in the production of *Nitrogen Mustard, HN–1*, which is obsolete. Nevertheless, it is of interest to describe this Spec

REQUIREMENTS AND TESTS:

1) *Appearance.* It shall be a clear, orange to light straw in color, liquid, free from all visual suspended matter or deposit. A composite sample shall be examined visually

2) *Specific Gravity at 20/20°* – 1.015 to 1.025g/cc. It shall be detd by means of a pycnometer in accordance with Method 4183 of Federal Test Method Standard No 141

3) *Nitrogen Content* – 10.36 to 10.57%. It shall be detd by Kjeldahl Method. Weigh in a thin glass bulb ca 0.4g of material to the nearest 0.1mg. Place it in a 500-ml Kjeldahl flask, add 10ml of concd sulfuric acid and cool in an ice bath. Break the bulb with the flattened end of a long glass rod making sure that the bulb and its stem are completely crushed. Rinse the rod with 20ml of concd sulfuric acid, add 10g of powdered K_2SO_4 (or anhydrous Na_2SO_4) and 0.5g of powdered $CuSO_4$ (or 0.3g of selenium) or a suitable combination of the catalyst

Note: Mercuric oxide, 0.78g, or an equivalent amt of Hg may replace copper sulfate or Se, but in this case Na_2S must also be added later to remove the mercury before the distillation, thus preventing the formation of mercury-amido compds which are not completely decompd when treated later by NaOH soln

Next, add 20ml of sulfuric acid, pouring it slowly down the neck so that any of the sample adhering to the neck of the flask is washed down. Incline the flask at an angle of ca 45° and gently heat over a free flame or electrically until frothing has ceased. Increase the heat until the acid boils briskly and continue heating until the liquid either acquires a clear green color or becomes nearly colorless. This takes about 30 mins. Cool and cautiously add 150ml of water, cool again, and then add 100ml of a 30% NaOH soln poured down the side of the flask so that it does not mix with the acid soln. Add a few pieces of granulated Zn and connect the flask, at once, by means of a rubber stopper, to the Kjeldahl distillation apparatus

Note: An alternate procedure is to add the alkali thru a small separatory funnel passing thru a 2nd hole in the stopper. No loss of ammonia can result with this method

Then mix the layers carefully by rotating the flask, and distil ca 200ml into a 500ml flask contg 50ml of 0.1N hydrochloric acid, and 6 drops of indicator soln (0.9g of Na alizarinsulfonate with 0.125g of indigo carmine dissolved in 100ml of water). Rinse the adapter into the receiving flask and titrate the excess acid with standardized 0.1N NaOH to the gray transition point of the indicator (pH=5.4). Calculate as follows:

$$\% \text{ Nitrogen} = \frac{1.401\,(AB-CD)}{W}$$

where:

A = ml of HCl
B = normality of HCl
C = ml of NaOH soln
D = normality of NaOH soln
W = weight of sample

4) *Water Content, as Received* – 0.20%, maximum. Determine its content in accordance with Method No 3001 of Federal Test Method Standard No 791

5) *Distillation Range* – 240° to 270° and *Total Distillate Between 240° & 265°* – 96.0% minimum

Use Method 1001 of Federal Test Method Standard No 791, except that, due to the high viscosity of the material, cooling water at 40–45° shall be substituted for the ice-water in the condenser

6) *Rejection Criteria.* If composite specimen fails to conform to this specification, the lot represented shall be rejected

Ethyldimethylanilines or **Ethylaminoxylenes.** See N-Ethylxylidines in this Vol

N-Ethyl-(dimethylphenyl)-amine or N-Ethyldimethylaniline and Derivatives (Äthyl-vic-o-xylidin in Ger),

N-Ethyl-2,3-dimethylaniline, $(CH_3)_2C_6H_3.NH.C_2H_5$; mw 149.24, liq, bp 227–28°. It is prepd by hydrolysis of the corresponding acetamide with 40% H_2SO_4

N-Ethyl-2,6-dimethylaniline (Äthyl-vic-m-xylidin in Ger), mw 149.24, bp 217–18°, oil smelling of camphor. It is prepd by reacting vic-m-xylidine with EtBr and NaOH at 180°

N-Ethyl-2,5-dimethylaniline (Äthyl-p-xylidin in Ger), mw 149.24, bp 222–23° (748 torr), oil smelling of naphthalene. It is prepd by hydrolysis of the corresponding benzene sulfonate with concd HCl at 150°

Ref: Beil **12**, 1101, 1109, 1137

N-Ethyl-2,3-dimethyl-4,6-dinitroaniline, $(CH_3)_2C_6H(NO_2)_2.NH.C_2H_5$; mw 239.23, N 17.57%; orn ndls, mp 75–6°. It is prepd from ethylamine and the 3,4,6-trinitro compd
Refs: 1) Beil **12**, (479) 2) A.W. Crossley & W.R. Pratt, JChemSoc **103**, 987(1913)

N-Ethyl-3,4-dimethyl-2,6-dinitroaniline, mw 239.23, orn ndls, mp 115°, sol in benz, chlf; prepd from ethylamine and the 3,4,5-trinitro compd
Refs: 1) Beil **12**, (481) 2) A.W. Crossley & W.R. Pratt, JChemSoc **103**, 986(1913)

N-Ethyl-2,5-dimethyl-4,6-dinitroaniline, mw 239.23, mp 133°; prepd from ethylamine and the trinitro compd
Refs: 1) Beil **12**, 1141 & [618] 2) J.J. Blanksma, RecTravChim **24**, 51(1904)

N-Ethyl-3,5-dimethyl-2,4,6-trinitroaniline, $(CH_3)_2C_6(NO_2)_3.NH.C_2H_5$; mw 284.23, N 14.78% ($NO_2$), mp 122°; prepd from ethylamine and the corresponding methoxytrinitro compd
Refs: 1) Beil **12**, 1133 2) J.J. Blanksma, RecTravChim **21**, 331(1902)

N-Ethyl-N-nitro-3,6-dimethyl-2,4-dinitroaniline [Ethyl-(dinitrodimethylphenyl)nitramine], $(CH_3)_2C_6H(NO_2)_2.N(NO_2).C_2H_5$; mw 284.23, N 14.78% ($NO_2$), cryst (from alc, HNO_3 or acet ac), mp 36°. It is prepd by nitration of the already ring nitrated compd
Refs: 1) Beil **12**, 1141 2) J.J. Blanksma, RecTravChim **24**, 51(1904)

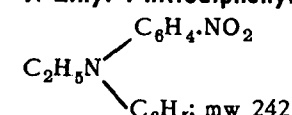
N-Ethyl-N-nitro-3,5-dimethyl-2,4,6-trinitroaniline [Ethyl-(trinitrodimethylphenyl)nitramine], $(CH_3)_2C_6(NO_2)_3.N(NO_2).C_2H_5$; mw 329.24, N 17.02% ($NO_2$), colorless crysts, mp 85°. It is prepd by nitration of the already ring nitrated compd
Refs: 1) Beil **12**, 1134 2) J.J. Blanksma, RecTravChim **21**, 331(1902)

2-Ethyl-1,3-dimethyl(propanediol). See 1,1-Di(methylol)-propane in Vol 5, p D1358-R

Ethyldiphenylamine and Derivatives

N-Ethyldiphenylamine, $C_2H_5.N(C_6H_5)_2$; mw 197.18, liq, sp gr 1.048, bp 295–97°; 150° (10mm); insol in w, sol in alc. It can be prepd by reacting EtBr with diphenylamine and KNH_2
Ref: 1) Beil **12**, 181 & [105]

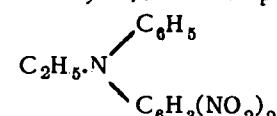
N-Ethyl-1-nitrodiphenylamine,



$C_2H_5N(C_6H_4.NO_2)(C_6H_5)$; mw 242.27, N 11.56%; orn blades, mp 50–51°. It is prepd from 1-nitrodiphenylamine, ethyl sulfate and base
Refs: 1) Beil, not found 2) F.R. Stoirie & S.H. Tucker, JChemSoc **1931**, 2255–63 & CA **26**, 452(1932)

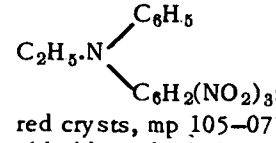
N-Ethyl-4-nitrodiphenylamine, mw 242.18, yel, mp 87–88°. It is prepd from 4-nitrodiphenylamine, ethyl iodide and base
Refs: 1) Beil, not found 2) K.A. Nuridzhanyan & V.A. Izmail'skii, ZhurVsesoyuzKhimObshchestva Im D.I. Mendeleeva **5**, 237–8(1960) & CA **54**, 20927(1960)

N-Ethyl-2,4-dinitrodiphenylamine,



$C_2H_5.N(C_6H_5)(C_6H_3(NO_2)_2)$, mw 287.28, N 9.75% ($NO_2$); mp 95°(97.5°), sp gr^{10} 1.364, red prisms (alc); sol in HAc, alc, ligroin, benz; sl sol in hot w & eth. It is prepd from ethyl aniline and 4-chloro (or bromo)-1,3-dinitrobenzene with NaAc (or boiling in alc)
Refs: 1) Beil **12**, 753, (362) 2) J. Forrest et al, JCS **1946**, 454–6 & CA **41**, 405b(1947)

N-Ethyl-2,4,6-trinitrodiphenylamine,



$C_2H_5.N(C_6H_5)(C_6H_2(NO_2)_3)$; mw 333.085, N 12.62%; red crysts, mp 105–07°. It is prepd from picryl chloride and ethyl aniline in alc
Refs: 1) Beil **12**, 766 & (370) 2) A. Hantzsch, Ber **43**, 1678(1910)

N-Ethyl-hexanitrodiphenylamine or **N-Ethyldipicrylamine,** $C_2H_5.N:[C_6H_2.(NO_2)_3]_2$, mw 467.27, N 20.98%, OB –70.2%, wh ndls, mp 198–200°, 201–202°, sol in acetone & EtAc; prepd from reaction of nitric acid with N-Et-N-phenyl-2,4-dinitroaniline
Refs: 1) Beil **12**, 766 2) A. Mulder, RecTravChim **25**, 122(1906)

N'-Ethyl-N,N-diphenylurea. See Acardite III (Ethylacardine) in Vol 1, p A8-R

ETHYLENE AND DERIVATIVES

Ethylene (Ethene or Elayl), $H_2C{:}CH_2$; mw 28.05; colorless, flammable, dangerous to handle gas with characteristic sweet odor and taste; sp gr 0.975 (air = 1.0), mp −169.4°, bp −103.8°, flash p −136°C; explosive limits in air, % by vol, lower 3.0 & upper 34.0; sl sol in w, more in alc; sol in eth. Ethylene is a major component of petroleum refinery gas from cracking units, and is sometimes recovered therefrom by distillation or other means. Some pure ethylene is produced by passing hot ethanol vapors over a catalyst, such as activated alumina (Ref 4). Its laboratory prepn consists of heating ethanol in definite proportions with sulfuric acid of certain concns. By using a 90% acid and 90% alc, ethylene can be produced in a regular stream at a yield of 84 to 85% of theory (Ref 2). Accdg to footnote 11 in Ref 2, p 212, ethylene was manufd in the US by passing ethanol vapor over coke wet with phosphoric acid in towers Uses: The source of many ethyl and ethylene compds, such as synthetic ethanol, ethyleneglycol, styrene; also in oxyethylene welding and cutting of metals (Refs 4 & 5)

Kume (Ref 3) obtd on heating to 245° and at 1600kg/sq cm pressure of liquid ethylene (contg 0.02 to 0.06% oxygen) a polymerization product, which was white, spongy, wax-like solid melting at 107–10°. Heating too rapidly could produce an expln

Refs: 1) Beil **1**, 180, (75) & [149] 2) Naoúm, NG(1928), 212 3) T. Kume, RevPhysChem-Japan, ShinkichiHoriba Commem Vol **1946**, 98–107 & CA **44**, 1745(1950) 4) CondChem-Dict (1961), 464 5) Kirk & Othmer **8**,499–522 (1965) & **5**, 310–314(1971)

Ethylene-Carbon Tetrachloride Explosions. Accdy to Bolt (Ref 1), ethylene (5.6 moles) was gradually introduced into a 2600-ml autoclave contg 17.6 moles carbon tetrachloride and 0.45 moles benzoyl peroxide, serving as reaction initiator. The pressure was kept below 2500psi. After about 2 hours an expln occurred at 67°, discharging most of the reactants thru the ruptured safety disk

Note: A previous alkylation of CCl_4 with 1,3-butadiene was performed without mishap

Joyce (Ref 2) described three examples of uncontrolled reactions between ethylene and CCl_4, two of them resulting in violent explns. In each case benzoyl peroxide was the initiator. Precautions essential for avoiding explns appear to be restriction of reaction rate and adequate dissipation of the heat of reaction. The quantity of reaction initiator should be kept at a minimum.

Refs: 1)R.O. Bolt, Chem Engrg News **25**, 1866 (1947) & CA **41**, 5724(1947) 2)R.M. Joyce, Ibid 1866–67 (1947) & CA **41**, 5724 (1947)

Ethylene Chloride. See Ethylene Dichloride

Ethylene Dichloride; 1,2-Dichloroethene or Dutch Oil, $ClCH_2CH_2Cl$; mw 98.97; col oily liquid; sp gr 1.2554 at 20/4°, n_D 1.444, fr p 35.5°, bp 83.5°, flash p 70°F; sl sol in W; v sol in alc, eth & chlf. Was first propd in 1795 by Dutch chemists; prepd now by the action of chlorine on ethylene with subsequent distn; its industrial method of prepn was patented in 1915 in Germany (Refs 2 & 3)

It is stable in the presence of water, alkalies, acids, or actively-reacting chemicals and is resistant to oxidation. Will not corrode metals. Its vapors are poisonous

Used as solvent for fats, oils, waxes, cellulose esters and ethers, rubber, "Bakelite", resins, gums and other products; also as scavenger for TEL (tetraethyl lead) in gasoline

Considerable amt was produced in Germany during WWII, because on mild saponification (such as with Na bicarbonate) it gave ethyleneglycol, while saponification with alkalies produced vinyl chloride, and finally acetylene (Refs 3&4)

Refs: 1)Beil **1**, *84, (24)* & [52] 2)Th. Godschmidt & O.Matter, GerP298931(1915) 3) Ibid, 299074(1916) 4)Naoúm, NG (1928), 213-14 5)Cond Chem Dict (1961),467 6) Kirk & Othmer **5** (1964), 149-54

Ethylene(Ethane)Explosive Decomposition. Workers in Holland have observed that C_2H_4 under certain conditions and especially in the presence of $AlCl_3$ as catalyst and finely divided Ni as promoter, may decomp vigorously or even explode into its elements. It has been found that some solvents for $AlCl_3$ promote

polymerization of C_2H_4. The following cmpds have been involved in expln of C_2H_4 at temps and pressures of experiments described in Ref: C_5H_{12}, CH_3Cl and CH_3NO_2
Ref: H.A. Waterman et al, J Inst Petroleum **33**, 254-55 (1947) & CA **41**, 6721 (1947)

Ethylene Reaction with NO_2. Investigation at the laboratory of US Rubber Co (Ref 2) showed that when ethylene was treated with N_2O_4 by the method of Levy & Scaife (Ref 1), the following compds were obtd: 1,2-Dinitroethane, Nitroethylnitrate and Nitroethanol

A process is described for producing aliphatic nitrates or nitro compds from the corresponding nitrites or nitrose compds by oxidation with NO_2 or O and N_2O_2 or NO. Thus, pure dry CH_2: CH_2 and 15% of its vol of O were passed into liq NO_2 over a period of 7 hrs, the reaction mixt was withdrawn, degassed, and poured into cooled MeOH. Solid $O_2N.CH_2:CH_2.NO_2$ was thrown out and separated by filtration. The MeOH was removed from the filtrate, the residual oil treated on vacuum pump and extracted by a mixt of benz & water. The aq layer was neutralized, extracted with ether and the remainder distd to give 2-nitroethanol. The benz layer gave $O_2N.CH_2.CH_2ONO_2$ (Ref 3)
Refs: 1) N. Levy & C.W. Scaife, JChemSoc **1946**, 1096 & CA **41**, 2388 (1947) 2) USRubber Co, Quarterly Report **No 2** (Contract Nord 10219), p9 3) A.E.W.Smith, C.W.Scaife & ICI LTD, Brit P575604(1946); A.E.W.Smith, R.H.Staely, C.W.Scaife & ICI Ltd:, Brit P575618(1946); and C.W.Scaife, H. Baldock, A.E.W. Smith & ICI Ltd, Brit P586344(1947) & CA **41**, 6893-94(1947)

Ethylene Azide or Vinyl Azide. See Azidoethylene in Vol 1, pA638-L

Ethylene Nitrite See Ethylene Dinitrite

Ethylene Nitrate. See Ethylene Dinitrate or Ethyleneglycol Dinitrate

Nitroethylene (NEt) $H_2C:CH.NO_2$; mw 73.05, N19.17%; lt yel-green liq with lachrymatory props and sharp penetrating odor; sp gr 1.073 ag 13.8°, bp 39° at 70mm or 98.5° at 745mm; sol in water and in some organic solvents. It was prepd in small yield by Wieland & Sakelarios (Ref 2) using direct dehydration of 2-nitroethanol by means of anhydrous Na bisulfate. Much better results were obtd by thermal decompn of 2-Nitroethanol benzoate, when carried out in a Claissen flask at 180-230°. The flask was covered with friction tape to exclude light. Buckley & Scaife prepd it by dehydrating 2-Nitroethanol with phthalic anhydride (Ref 3). Nema (Ref 4) prepd it from CH_3NO_2+HCHO, thru intermediate formation of $O_2N.CH_2.CH_2.OH$, followed by dehydration with $KHSO_4$ as a catalyst. This gave a liquid compd, but if dehydration were done with H_3PO_3 as catalyst polymerization took place to form a tough solid. Boileau & Runavot (Ref 5) described a laboratory method using activated alumina and silica gel for dehydration. NEt easily forms a polymer, which is described below
Refs: 1) Beil **1** (81) 2) H. Wieland & E. Sakelarios Ber **52** 898-901(1919) & CA **13** 2874 (1919) 3) G.D. Buckley & C. W. Scaife, Brit P595282(1947) & CA **42**, 3773(1948); J Chem Soc **1947**, 1471-72 (1947) & CA **42**, 4907 (1947) 4) K. Noma, Chem High Polymers (Japan) **5**, 99-103 (1948) & CA **46**, 4471 (1952) 5) J. Boileau & Y. Runavot, MP **35**, 39-40 (1953) & CA **49**, 13886 (1955), 6) J.F.Brown, Jr. JACS **77**, 6341-51 (1955) & CA **50**, 2297 (1956) (IR spectrum) 7) L. Horner et al, Ann **591**, 108-17 (1955) & CA **50**, 2495 (1956) (Expl polymer)

Nitroethylene Polymer (NEtPol) $[H_2C:CH.NO_2]_x$; mw $[73.05]_x$, N 19.17%, OB to CO_2-7%; wh amorphous solid, mp-did not melt, but decompd slowly at 75°, igniting after 2 hours of heating; being heated at 100°, it deflagrated with evoln of a brown sooty smoke; when placed on a hot plate heated at 200°, it spontaneously decompd without expln. Small amts dropped on a red hot plate burned with a gentle puff. In the folds of comb-

ustible paper, it burned rapidly and smoothly. When wrapped in tin foil and hammered on an iron base, it did not explode. Qualitative capillary tests in a sealed tube indicated that it is a weak explosive

NEtPol is very sl sol in w; more sol in some org solvents including cyclic ketones; sol in hot alkalies forming a yel liquid; insol in dimethylformamide

It was prepd by Wieland & Sakellarios (Ref 2) by emulsifying monomeric 2-NEt in an aqueous medium (neutral or slightly alkaline). A similar method was used by Buckley & Scaife (Ref 4). Blatt (Ref 3) lists a method of prepn from 1-chloro-2-nitroethane and Na bicarbonate in 76% yield, developed during WWII in USA. Noma (Ref 5) prepd the polymer from Nitromethane and formaldehyde thru intermediate formation of Nitroethanol, followed by dehydration with H_3PO_3 catalyst

Following is laboratory method of prepn developed during WWII in USA: Agitate mechanically a 3-neck 500-ml flask contg 300-ml of freshly distd w, 2g calgon (emulsifier) and 5 drops of ascaridole. After displacing the air in the flask, with O-free nitrogen, add gradually 10g of freshly prepd Nitroethylene and continue agitation for 2 hours. Filter off the coagulated solid material and dry. Yield 91%

Blatt (Ref 3) lists the following props of NEtPol:

Hygroscopicity at 25° - gains 2.6% at 90% RH and 14% at 100%

Impact Sensitivity - comparable with TNT

International Test at 75° - ignites after 105 mins

Power by Ballistic Mortar Test - 29% of TNT

Thermal Stability at 100° - deflagrates in 23 mins

Uses - no information

Refs: 1) Beil **1**, (82) 2) W. Wieland & E. Sakellarios, Ber **52**, 901 (1919) & CA **13**, 2874(1919) 3) Blatt, OSRD **2014**(1944) 4) G.D.Buckley & C.W. Scaife, J ChemSoc **1947**, 1471-72 & CA **42**, 4907 (1947) 5) K. Noma, Chem High Polymers (Japan) **5**, 99-103 (1948) & CA **46**, 4471 (1952)

Nitrosoethylene, $H_2C{=}CHNO$; mw 57.05, N24.56%. Hypothesized as intermediate in the reaction of ethylene with NO_2 (Ref 2)

Refs: 1) Beil, not found 2) T.Takeuchi, Shokubai No **8**, 37-46 (1952) & CA **47**,11916 (1953)

Ethylene Dinitrite. Same as Ethyleneglycol Dinitrite or Glycol Dinitrite, described under Ethyleneglycol and Derivatives

Ethylene Dinitrate. Same as Ethyleneglycol Dinitrate or Glycol Dinitrate, described under Ethyleneglycol and Derivatives

Ethyleneamine. The name given in Cond ChemDict (1961), 464 to Diethylenediamine or Piperazine described in Vol 5,pp D1230-R & D1231. Compare with Ethylenediamine

Ethyleneaminoöxime and Derivatives

Ethyleneaminoöxime, $H_3C.C(NH_2){:}NOH$; mw 74.08, N 37.81%; ndls (alc + eth), mp 135° (dec), sol in water, alc; insol in eth & chlf; unstable. It is prepd from hydroxylamine and acetonitrile with NaOEt (Ref 2)

Refs: 1) Beil **2**, 188 2) E. Nordmann, Ber **17**, 2746(1884)

Nitroethyleneaminoöxime,
$(O_2N)H_2C.C(NH_2){:}NOH$; mw 119.08, N 35.29%; yel solid, mp – decomp violently when heated to about 108°. It can be prepd by action of Amm nitroacetonitrile on $NH_2OH.HCl$

Refs: 1) Beil **2**, 227 & (100) 2) W. Steinkopf & L. Boehm, Ber **41**, 1044–52(1908) & CA **2**, 1962(1908)

Ethylenebisacetamide and Derivatives

Ethylenebisacetamide (Called Diacéthylethylène diamine by Franchimont), $H_3C.CO.NH.CH_2{-}CH_2.NH.CO.CH_3$; mw 144.17, N 19.43%; crysts, mp 172–73°; sol in alc & water. It was first prepd by Linnemann in 1870 (Ref 1) by heating acetamide with acetic anhydride for 6 hrs at 250°. Later Franchimont & Dubsky (Ref 2) prepd it by heating to boiling the above substances in the presence of metallic Na. Bachmann (Ref 3) prepd it by adding 750ml acetic anhydride to 280ml of 64% aqueous ethylenediamine with stirring and cooling. The resulting clear soln was concentrated on a steam bath, while passi

a current of air, and then mixed with an equal volume of acetone and cooled. The pptd crysts (first crop, 403g) had mp 172–73°, while the 2nd crop (112g) was of lesser purity

Nitration gave the dinitroderivative described below

Refs: 1) Beil **4**, 253 & (416) 2) A.P.N. Franchimont & J.V. Dubsky, Rec **30**, 184(1911) 3) W.E. Bachmann et al, JACS **72**, 3133(1950) & CA **44**, 10656(1950)

N,N'-Dinitroethylenebisacetamide, $H_3C.CO.N(NO_2).CH_2–CH_2.N(NO_2).CO.CH_3$; mw 234.17, N 23.93%; col plts, mp 135.5–136° (decomp); (132–133° for crude product); solubilities – not given. It was prepd by Bachmann et al in 83% yield in the following manner: Prepare the nitrating acid just before use by slowly adding with stirring 40ml of acetic anhydride to 40ml of 98% nitric acid cooled in an ice-salt bath to 5° or below. Add gradually to this mixture (while stirring it and keeping at 5° or below) 15g of ethylenebisacetamide. Continue stirring for 5 hours keeping at 5° or below and then pour the mixture into 250ml of ice-water. Separate the ppt by filtration, wash it with water and crystallize from acetone. This should give about 20.1g of Dinitroethylenebisacetamide

When 10g of dinitrocompd was stirred with 27ml of concd Amm hydroxide, the EDNA (Ethylenedinitramine) was obtd in 91% yield

Refs: 1) Beil, not found 2) W.E. Bachmann et al, JACS **72**, 3133(1950) & CA **44**, 10656(1950)

N,N'-Ethylene-bis(anthranilonitrile) and Derivatives

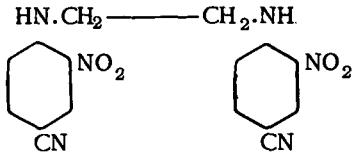
N,N'-Ethylene-bis(anthranilonitrile),

$CH_2NH.C_6H_4.CN$
|
$CH_2.NH.C_6H_4.CN$; mw 270.27, N 23.68%

Refs: 1) Beil, not found 2) CA, not found

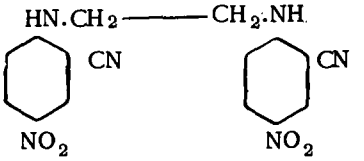
N,N'-Ethylene-bis(nitroanthranilonitrile), $C_{16}H_{12}N_6O_4$; mw 352.30, N 23.86%. Two isomers are known:

N,N'-di(2-Nitro-4-cyanophenyl)-ethylenediamine (Blanksma) or *4,4'-Ethylenediimino-bis(3-nitrobenzonitrile)* (CA Index),



yel crysts, mp >360°; prepd by heating a soln of 1-chloro-2-nitro-4-cyanobenzene & ethylenediamine hydrate in alc for 5 hrs in a sealed tube. The reaction product was allowed to stand for several hrs with a lukewarm soda soln, from which the pure product was separated (Ref 2)

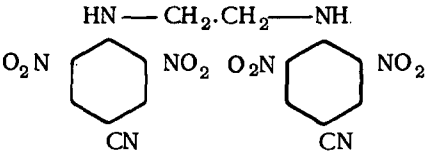
N,N'-bis(2-cyano-4-nitrophenyl)-α,β-diaminoethane (Hartmans) or *N,N'-Ethylene-bis(5-nitroanthranilonitrile)* (CA Index),



yel crysts having a sandy taste, mp >360°; was prepd by heating 1-chloro-2-cyano-4-nitrobenzene & α,β-diaminoethane in alc in a sealed tube for 3 hrs. On cooling the cryst product was obtd (Ref 3)

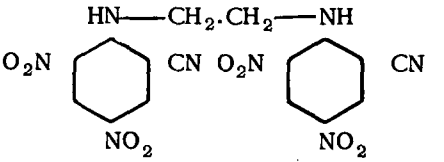
N,N'-Ethylene-bis(nitroanthranilonitrile), $C_{16}H_{10}N_8O_8$; mw 442.30, N 25.34%. Two isomers are known:

N,N'-di(2,6-Dinitro-4-cyanophenyl)-ethylenediamine (Blanksma) or *4,4'-Ethylenediimino)-bis(3,5-dinitrobenzonitrile)* (CA Index),



yel crysts, mp 282°; was prepd when a soln of 1-methoxy-2,6-dinitro-4-cyanobenzene & ethylenediamine hydrate in alc was boiled with a reflux condenser for 3 hrs on a water bath. On cooling the product precipitated (Ref 2)

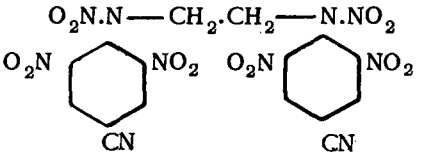
N,N'-bis(2-Cyano-4,6-dinitrophenyl)-α,β-diaminoethane (Hartmans) or *N,N'-Ethylene-bis(3,5-dinitroanthranilonitrile)* (CA Index)



yel tasteless crysts (from alc), mp 239°; was prepd when a mixt of 1-methoxy-2-cyano-4,6-dinitrobenzene and α,β-diaminoethane in alc was boiled under reflux for 3 hrs. On cooling the product separated (Ref 3)

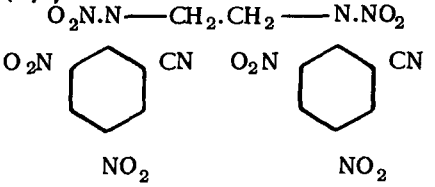
N,N′-Ethylenedinitramine-bis(nitroanthranilonitrile), $C_{16}H_8N_{10}O_{12}$; mw 520.29, N 26.92%. Two isomers are known:

N,N′-di(2,6-Dinitro-4-cyanophenyl)-ethylenedinitramine (Blanksma) or **4,4′-[Ethylenebis(nitroimino)]-bis(3,5-dinitrobenzonitrile)** (CA Index)



colorless plates (from acet + alc), mp 203–04° (dec); when heated in a sealed capillary in a flame, the compd exploded. It was prepd by nitrating with anhyd nitric acid the above 2-nitro deriv or the 2,6-dinitro deriv. A mixed mp of the two products showed the substances to be identical (Ref 2)

N,N′-bis(2-Cyano-4,6-dinitrophenyl)-α,β-dinitroethane (Hartmans) or **N,N′ Ethylene-bis-(N,3,5-trinitroanthranilonitrile)** (CA Index)



yel tasteless crysts (from alc), mp 145°; was prepd by nitrating the 2-cyano-4-nitro deriv above with abs nitric acid. Nitration of N,N -bis(2-cyano-4,6-dinitrophenyl)-α,β-diaminoethane with abs nitric acid gave lt yel crysts melting at 218°, indicating a purer product

Refs: 1) Beil, not found 2) J.J. Blanksma & C.H.D. Witte, Rec **60**, 818–20(1941) & CA **37**, 3073(1943) 3) H.M.A. Hartmans, Rec **65**, 470–72(1946) & CA **41**, 734(1947) 4) Not found in later refs thru 1966

2,2′-Ethylene-bis(4,6-diamino-5-triazene) or **Succinoguanamine,**

$[N{=}C(NH_2)N{=}C(NH_2)N{=}C.CH_2\frac{}{}]_2$; mw 248.25, N 56.42%; mp over 335°. It was prepd by reacting diethyl succinate with biguanide in 75% yield

Refs: 1) Beil, not found 2) J.T. Thurston, USP 2427315(1947) & CA **42**, 1972(1948)

Ethylene-bis(N,N-diphenylurea) or **Bis(diphenylcarbamyl) -ethylenediamine,**

$(C_6H_5)_2$:N.CO.NH.CH_2.CH_2.NH.CO.N:$(C_6H_5)_2$; mw 450.52, N 12.44%; crysts (from chlf), mp 187°; insol in w; soly in chlf ca 20% at 25°. It can be prepd by refluxing diphenylcarbamylchloride and ethylenediamine in ethanol. Heat of Combstn at C_v 3479.05kcal/mol and at C_p 3481.08; Heat of Formation at C_v 44.73 and at C_p 54.01kcal/mol. It may be suitable for use in proplnts

Refs: 1) Beil, not found 2) R. Lévy, MP **32**, 309(1950) 3) P. Tavernier & M. Lamouroux, MP **38**, 73 & 83(1956)

Ethylenebis(guanidine) and Derivatives

1,1′-Ethylenebis(guanidine),

CH_2.NH.C(NH_2):NH
|
CH_2.NH.C(NH_2):NH; mw 144.18, N 58.29%. It was prepd as various salts by reacting ethylenediamine hydrate with 5-methyl-isothiourea salts (Ref 1). The **dinitrate** forms ndls, dec at 252°. The **dipicrate** dec at 284–85°

1,1′-Ethylenebis(3-nitroguanidine),

$[O_2N.NH.C(=NH)NH.CH_2\frac{}{}]_2$; mw 234.18, N 47.85%, OB –61.5%; mp 248–50°; prepd in 76% yield by reacting ethylenediamine with 2-methyl-3-nitro-2-thiopseudourea

Refs: 1) Beil **4**, [693] 2) L. Fishbein & J.A. Gallaghan, JACS **76**, 1877–9(1954) & CA **49**, 6838(1955)

2,2′-(Ethylenebisimino)-diethanol and Derivatives

2,2′-(Ethylenebisimino)-diethanol or *2,2′-(Ethylenediimino)-diethanol,*

$(HO.CH_2.CH_2NH.CH_2\frac{}{})_2$; mw 148.21, mp 102–3°; prepd by reacting 2-hydroxyethylamine with a dihaloethane compd (Ref 2)

Refs: 1) Beil, not found 2) I.G. FarbenindAG, FrP 801121(1936) & CA **31**, 111(1937)

2,2′-(Ethylenebisimino)-diethanol Dinitrate or *2,2′-(Ethanoldiimino)-diethanol Dinitrate,*

$(ONO_2.CH_2.CH_2.NH.CH_2\frac{}{})_2$; mw 238.20, N 11.76%($NO_2$). Prepn not found in open literature

Refs: 1) Beil, not found 2) R.N. Jones & G.D. Thorn, CanJRes **27B**, 828–60(1949) & CA **44**, 2848(1950)(UV spectrum)

2,2'-(Ethylenebisnitroimino)-diethanol or *2,2'(Ethylenedinitroimino)-diethanol*, $(HO.CH_2.CH_2.N(NO_2)CH_2\rightarrow_2$; mw 238.20, N 11.76% ($NO_2$), crysts. It was prepd by reacting ethylenediaminedinitramine (EDNA) with ethylene oxide and NaOH (Ref 2)

Refs: 1) Beil, not found 2) J.R. Johnson et al, USP 2683165(1955) & CA **49**, 7590(1955)

2,2'-(Ethylenebisnitroimino)-diethanol Dinitrate or 2,2'-(Ethylenedinitroimino)-diethanol Dinitrate, $(ONO_2.CH_2.CH_2.N(NO_2)CH_2\rightarrow_2$; mw 328.20, N 17.07%, OB −39.0%, crysts, mp 66.5–67.5°. It was prepd by nitration of the above 2,2'-(Ethylenebisnitroimino)-diethanol

Refs: 1) Beil, not found 2) J.R. Johnson et al, USP 2683165(1955) & CA **49**, 7590(1955)

(Ethylenebisimino)-dimethanol and Derivatives

(Ethylenebisimino)-dimethanol or *(Ethylenediimino)-dimethanol.* Same as N,N'-Di(methylolamino)ethane, described in Vol 5, p D1351-L

(Ethylenebisnitroimino)-dimethanol or *(Ethylenedinitroimino)-dimethanol.* See N,N'-Dimethylol-1,2-dinitroiminoethane in Vol 5, p D1351-L

(Ethylenebisnitroimino)-dimethanol Dinitrate or **(Ethylenedinitroimino)-dimethanol Dinitrate.** See N,N'-Dimethylol-1,2-dinitroiminoethane Dinitrate in Vol 5, p D1351-L

(Ethylenebisimino)-imidazolidine and Derivatives

(Ethylenebisimino)-imidazolidine, $\leftarrow CH_2.N.CH{=}N.CH_2.CH_2\rightarrow_2$; mw 166.21, N 33.71%. Not found in literature

Refs: 1) Beil, not found 2) CA, not found

1,1'-[Ethylenebis(3-nitro-2-nitroimino)]-imidazolidine, $\leftarrow CH_2.NC(=N.NO_2)N(NO_2)CH_2.CH_2\rightarrow_2$; mw 376.25, N 14.89% ($NO_2$), OB −59.6%, mp 177–78° (dec). It was prepd in 96% yield by nitration of the corresponding 2-nitroimino compd

Refs: 1) Beil, not found 2) A.F. McKay et al, CanJChem **29**, 382–90(1951) & CA **46**, 7094 (1952)

Ethylenebisurea and Derivatives

1,1'-(Ethylenebisurea), (Ethylenebiscarbamide) or *Ethylenediurea,* $CH_2.NH.CO.NH_2$ | $CH_2.NH.CO.NH_2$; mw 148.15, N 37.82%; colorless crysts, mp 192–94°; sol in boiling water. It was first prepd in 1861 by Volhard (Refs 1 & 2) by interaction of AgCN with ethylenediamine hydrochloride. The compd was also prepd by Davis & Blanchard (Ref 3) and then later by Bachmann et al (Ref 4). They refluxed for 3 hours urea with 60% aqueous ethylenediamine, obtg the crude product of 188° in 89% yield; recrystallization from aqueous alc raised its mp to 192–93°

When treated with weak nitric acid it rendered the dinitrate, while strong nitric acid gave the dinitrocompd

1,1'-(Ethylenebisurea)Dinitrate, $\leftarrow CH_2.NH.CO.NH_2.HNO_3\rightarrow_2$; mw 272.175, N 10.29% ($NO_2$), OB −35.3%, mp 150–51°. It was prepd by nitration of ethylenebisurea with dilute nitric acid

Refs: 1) Beil **4**, 254 & [693] 2) J. Volhard, Ann **119**, 394(1861) 3) T.L. Davis & K.C. Blanchard, JACS **51**, 1790(1929) & CA **23**, 3442(1929) 4) W.E. Bachmann et al, JACS **72**, 3133(1950) & CA **44**, 10656(1950)(Parent and dinitrate)

1,1'-[Ethylenebis(3-nitrourea)] or **3,3'-Dinitro-1,1'-ethylenebisurea,** $CH_2.NH.CO.NH.NO_2$ | $CH_2.NH.CO.NH.NO_2$; mw 238.14, N 35.27%, OB −40.3%, colorless ndls, mp 197–98°(dec). It was first prepd during WWII by workers of NDRC by nitrating ethylenebis(urea) with strong nitric acid (Ref 2). Bachmann et al (Ref 3) prepd it by dissolving 1.46g of ethylenebis(urea) in 3.6ml of 98% nitric acid, precooled to 0°, followed (after deposition of ppt) by drowning it in ice water. After allowing to stand for 15 mins, the ppt was filtered off and purified. The yield was 0.67g. The compd may also be prepd by drowning in concd sulfuric acid at −5° the dinitrate of ethylenebis(urea)

Its expl props were not reported, but its heat of combstn is given in Ref 2 as 540.4kcal/mol

Refs: 1) Beil, not found 2) A.H. Blatt & F.C. Whitmore, OSRD Rept **1085**(1942), p 34 3) W.E. Bachmann et al, JACS **72**, 3133–34(1950) & CA **44**, 10656(1950)

Ethylenebisurethane and Derivatives
Ethylenebisurethane, Ethylene-(1,2-diurethane) or *Juinite*, $(CH_2.NH.CO_2.Et)_2$; mw 204.23, N 13.72%; ndls, mp 112°, sol in alc & eth. It was prepd by reacting ethylenediamine with ethyl chloroformate and vacuum distillation purification. Its heat of combstn is 1066kcal/mol & heat of formation 231kcal/mol at const vol. It was used in some French proplnts
Refs: 1) Beil **4**, 254 & [693] 2) L. Médard & M. Thomas, MP **34**, 421 (1952) 3) P. Tavernier, MP **38**, 306 & 328 (1956)

Ethylenebisurethane-1-Azide; Ethylene-(1,2-diurethane)-1-Azide or **1-Azidoethylene-diurethane**, $CH_2.NH.CO_2.Et$
|
$CH(N_3)\cdot NH.CO_2.Et$; mw 245.24, N 28.56%; dark yel oil, changes to another material on storage. It was prepd by boiling azido succinylazide with alc
Refs: 1) Beil **4**, (450) 2) T. Curtius, Ber **45**, 1056, 1092 (1912)

Ethylenebromide. See Ethylenedibromide in this Vol

Ethylenebromohydrine. See 2-Bromoethanol in Vol 2, p B311-R

Ethylene Chloride or Vinyl Chloride. See Chloroethylene in Vol 3 of Encycl, p C255-R. Its 1-Nitro compd and Polymer of 1-Nitro compd are described on p C256-L

Ethylene Chlorohydrin. See Chloroethanol or Chloro Ethyl Alcohol in Vol 3, p C254. Its Nitrite, Nitrate, 2-Nitro- and 2-Nitro-nitrate-derivs are described on p C255

Ethylene Cyanohydrin or **β-Hydroxypropionitrile**, $HO.CH_2.CH_2.CN$; mw 71.08, N 19.71%; poisonous straw-colored liq, sp gr 1.0404 at 25°/4, fr p −46°, bp 227–28°(dec), vapor pressure 20mm at 117; miscible with w, acet, ethanol, chlf, methyl-ethyl ketone; sl sol in eth; insol in benz, CCl_4 & naphtha. It can be prepd by interaction of ethylene oxide with hydrocyanic acid. It is used as solvent for certain cellulosic esters and inorganic salts
Refs: 1) Beil **3**, 298, (113) & [213] 2) Cond-ChemDict (1961), 465-L

ETHYLENEDIAMINE AND DERIVATIVES

Ethylenediamine ("en"), 1,2-Diaminoethane, $H_2N.CH_2.CH_2.NH_2$; mw 60.10, N 46.61%; colorless, volatile liq; strongly basic; sp gr 0.8995 at 20/20°, mp 8.5°, bp 116–17°, n_D 1.4540 at 26°, vap press 10.7mm at 20°, viscosity 0.0154 poises at 25°, dielectric const 16.0 at 18°, fl p 43°(110°F), heat of combstn 452.6cal/mole, latent heat of fusion 77cal/g at 0°, latent heat of vaporization 167cal/g (calcd); very sol in w & alc; sl sol in eth; insol in benz; causes severe eye and skin burns

Typical specifications for commercial product: purity – not less than 66% by wt; boiling range at 760mm – below 115° none and above 122° none; odor – mildly ammoniacal and color – water-white

It can be prepd by heating ethylenedichloride and ammonia with subsequent distillation; purified by redistillation

It is remarkable for its ability to take part in the formation of coordination compds, such as *ammines* (See Vol 1 of Encycl, pp A275ff); some of these compds are expl, as, for example, copper complexes described here under "Ethylenediamine Complexes". On neutralization with nitric acid it forms the expl Ethylenediamine Dinitrate (See below), while under other conditions it gives the expl Ethylenedinitramine, which, however, is usually prepd by nitrating ethyleneurea. Other expl compds, such as Dichlorate, Diperchlorate are prepd from ethylenediamine

Ethylenediamine, as such, is used as a solvent for various compds, as a corrosion inhibitor in antifreeze soln, stabilizer in rubber latex, etc. Its use as a colorimetric reagent for expls, such as TNT, etc, is discussed in Vol 3 of Encycl, p C406-L
Refs: 1) Beil **4**, 230, (398), [676] & {478} 2) W.E. Bachmann, OSRD **152** or PBA 31100 (1941) (Prepn of expls from ethylenediamine) 3) A. Robertson, JSCI **67**, 221 (1948) (Thermal decompn of ethylenediamine) 4) GerPat Appl No F8313(12q, 1/01), translation Res-InfoServRept **96013**, Feb 9, 1952 & April

2, 1953 (Process for prepn of ethylenediamine from ethylenechloride and highly concd aqueous ammonia) 5) CondChemDict (1961),465-L 6) Kirk & Othmer (1965), 22–37 8) Sax, 3rd edition (1968), 743

Ethylenediamine Chlorate. See Ethylenediamine Dichlorate

Ethylenediamine-Chromium Tetroxide, $H_2N.CH_2.CH_2.NH_2+CrO_4+2H_2O$; greenish-grey crysts, mp – decomp with evoln of water and deflagration; nearly insol in w; is stable in the dark. It can be prepd from ethylenediamine, chromic acid and 30% hydrogen peroxide
Refs: 1) Beil **4**, 232 2) K.A. Hofmann, Ber **39**, 3182(1906)

ETHYLENEDIAMINE COMPLEXES

Most complexes described in the literature are those of copper, but there are also complexes of cobalt, such as:
Cobalt (III) Dichloro-bis-ethylenediamino Chlorate, $[Co(en)_2Cl_2]ClO_3$; Cobalt (III)-Dichloro-bis-ethylenediamino Perchlorate, $[Co(en)_2Cl_2]ClO_4$; Cobalt (III) Tris-ethylenediamino Trinitrate, $[Co(en)_3](NO_3)_3$

The following "en" copper complexes were prepd by Morgan & Burstall (Refs 1 & 2)
Bis-ethylenediaminocupric Persulfate, $[Cu(en)_2]S_2O_8$; purplish-red acicular crysts; mp – explodes on heating in air or while in concd sulfuric acid soln; it is unstable; detonates readily on percussion; sparingly sol in cold w, more readily in warm water. It can be prepd by adding aq K persulfate (1 mole) to an aq soln of Cu sulfate (1 mole), contg "en" (2 moles). Its expl props are comparable with those of Tetra-aquoethylenediaminocupric Perchlorate (Refs 2 & 3)

Tetra-aquoethylenediaminocupric Perchlorate, $[Cu(en).4H_2O]ClO_4$; dark bluish-violet ndls; mp – decomp quietly; explodes violently when heated with oxidizing agents; slightly hygroscopic but stable in air; sol in w; insol in alc and most org solvents. It can be prepd by concentrating an aq soln of ethylenediamine (1 mole) with cupric perchlorate (1 mole) (Refs 1 & 2)

Amiel (Ref 3) prepd several expl "en" complexes by the action of a 75% aq soln of "en" on $CuX_2.6H_2O$, where X=ClO_3, ClO_4 or NO_3; as the reaction is exothermic, it must be conducted cautiously with cooling to avoid explns

The compds of Amiel listed below were deliquescent, bluish-purple crysts sol in w, sl sol in acet and insol in alc or ether
Chlorates: $Cu(ClO_3)_2.2en.H_2O$ and $Cu(ClO_3)_2$-$3en.H_2O$; expl at 174°, and 178°, respectively; both also explode on impact and on contact with concd sulfuric acid
Nitrates: $Cu(NO_3)_2.2en$ and $Cu(NO_3)_2.3en.H_2O$; explode at 264° and 292°, respectively
Perchlorates: $Cu(ClO_4)_2.2en.H_2O$ and $Cu(ClO_4).3en.H_2O$; explode at 264° & 292°, respectively, as well as by impact (Ref 3a)

Of the above three complexes, the chlorates proved to be the most powerful and were recommended for use in primary compns

Cirulis & Straumanis (Ref 4) prepd the expl:

Ethylenediaminodiazido-copper(II), $[Cu(en)](N_3)_2$ or $[(N_3)_2Cu(en)]$; dk green ndls, mp 175–77°; expl mildly at 210° (on preheated block) or by impact of 1 kg weight (25cm); burns in flame with crepitation; sl sol in w; insol in alc or eth. It can be prepd by addn of Copper Azide to an amine soln
Refs: 1) G. Morgan & F. Burstall, JCS **1926**, 2026 & CA **20**, 3401(1926) 2) Ibid, JCS **1927**, 1259–69 & CA **21**, 3166(1927) 3) J. Amiel, CR **199**, 201–03(1934) & CA **28**, 5774(1934) 3a) P. Pfeiffer & H. Glaser, JPrChem **151**, 134–44(1938) & CA **32**, 8361(1938) 4) A. Cirulis & M. Straumanis, ZAnorgChem **251**, 347–48(1943) & CA **37**, 6574[3](1943)

Ethylenediamino Dichlorate. See Vol 2, p C188-R, under CHLORATES

Ethylenediamine-Diethylenetriamine Complexes. The literature contains numerous refs to the expl nature of certain coordination compds. Evidence demonstrates that metal compds contg a) coordinated ammonia & related N-contg donor molecules and b) coordinated and/or ionic groups of an oxidz nature such as perchlorate, chlorate, nitrate, nitrite (or nitrato- & nitro-groups) will decomp violently under various conditions. For this reason, due caution should be exercised in the prepn, handling and storage of compd defined above

Tomlinson et al (Ref 2) detd the expl props of many metal ammines and found tris-Ethylene-

diamine Cobalt (III) Nitrate & bis-Diethylenetriamine Cobalt (III) Perchlorate, among others, are expl in nature. These compds & other oxygenated compds, particularly those which are "oxygen balanced" should be considered exceedingly dangerous

Refs: 1) Beil **4**, 233, (400), [678] & {485} 2) W.R. Tomlinson, K.G. Ottoson & L.F. Audrieth, JACS **71**, 375–76 (1946) & CA **43**, 5187 (1944)

Ethylenediamine Dinitrate (EDD or EDAD) (Ger DIAMIN), (Designated by Franchimont as NEDA), $H_2N.CH_2.CH_2.NH_2.2HNO_3$ or $^-O_3N.NH_3^+-CH_2.CH_2-NH_3^+.NO_3^-$; mw 186.13, N 30.10%, OB to CO_2 –25.8%; wh crysts, sp gr 1.595 at 25/4°, mp 185–87°; sol in w, insol in alc or eth. It can be prepd by neutralization of ethylenediamine dihydrate with concd nitric acid, followed by concn and crystallization of salt

It forms eutectics with AN, but is immiscible with molten TNT

Its properties are as follows (Refs 2a, 3, 6, 7, 7a, 8 & 9):

Brisance – lower than for TNT

Combustion Test (French) – ignites by open flame after 1 min and continues to burn at the rate of 1.5cm/min

Detonation Velocity (by Dautriche Method): 4650m/sec at sp gr 1.00, 6270 at 1.33 and 6915 at 1.50

Gap Test – 3.75cm between paper cartridges each contg 50g sample at density 0.75

Heat of Combustion – 374.7kcal/mole (Ref 4a)

Heat of Explosion –127.9/159.3kcal/mole

Heat of Formation – 156.1kcal/mole

Hygroscopicity at 25° and 90% RH – gained 1.24%

Ignition Temperature (French Test) – ignites in 6 secs, when a small sample in test tube was plunged into a bath preheated to 370°; the time was 1 sec, when the temp in the bath was 430°

Impact Sensitivity – less sensitive than PA and TNT; FI 120% PA. French test with 10kg weight (mouton) gave at the height of drop 2.50 meters, 50% detonations

Initiation Sensitivity – 50g sample pressed to sp gr 1.23 in a paper cylinder 30mm diam required 2g MF for its detonation

Power (Strength) – sl more powerful than TNT, but less powerful than EDNA. Ballistic Mortar Test – 114% TNT. Trauzl Lead Block Test – 115% PA or 120–125% TNT

Rifle Bullet Test was detd by Médard & Cessat (Ref 7a) for charges of sp gr 1.0 – no detonations on impact of bullets with velocities below 960m/sec and incomplete detons for velocities around 1000m/sec

Sensitivity to Impact. See Impact Sensitivity

Sensitivity to Initiation. See Initiation Sensitivity

Stability – decomp with formation of brown fumes at 270°; does not expl at 360°

Thermal Stability. Robertson (Ref 5) stated that when heated in vacuo EDD vaporizes and condenses on cold walls without noticeable decompn. When heated at 10mm pressure in atmosphere of nitrogen or air, it undergoes autocatalytic decompn betw 230° & 360°

Thermal Stability at 135°. Accdg to Blatt (Ref 2a), it does not become acid and does not expl in 300mins

Uses – used by Germans during WWII as pressed charges in shells and as cast charges in eutectic mixts with AN. In mixts with wax it was used in boosters (Refs 3 & 10)

UV Spectra were detd in Canada (Ref 7)

Velocity of Detonation. See Detonation Velocity

Ethylenediamine Dinitrate was manufd in Germany, under the name of DIAMIN, from synthetic raw materials: ethanol, ammonia and nitric acid (Ref 3)

Its uses during WWII by the Germans are indicated above under Use s

Two of its German eutectic mixtures were investigated at PicArsn (Ref 3). They were: 1) EDD 45 & AN 55% and 2) EDD 45, AN 53.5 & Al 1.5%

Their props were approximately as follows: *Ballistic Strength* 126% TNT; *Brisance* – less than for TNT; *Decomposition Temperature* – 278°C; *Detonation Velocity* – less than for EDD; *Impact Sensitivity* – similar to that of EDD; *Reaction with Metals* – similar to that with Amatols (See Vol 1 of Encycl, p A161-R)

Ficheroulle (Ref 6), proposed a mixture with favorable OB to CO_2 consisting of EDD 42.8 & AN 57.2% and designated as **NB79.** Its props are in comparison with *Schneiderite* (DNN 87–88 & AN 13–12%): *Detonation Velocity* – 6183m/s at d 1.35 (Sch 5723); *Gap Test* – 7.75cm betw two paper cartridges contg 50g sample at d 0.94 (Sch 6.5cm); *Impact Test with 5kg Weight* – slightly more sensitive than Sch;

Initiation Sensitivity – 0.20g MF for sample at d 0.94; *Power by CUP* (Modified Trauzl Test) – 113.5% PA (Sch 113%); *Stability at 100°* – turns litmus paper red in 43.5 hrs (Sch 28.75 hrs). Although its expl props and stability are slightly more favorable than those of Schneiderite, it is not recommended by Ficheroulle as a bursting chge of projectiles in lieu of Schneiderite due to its higher cost

LeRoux (Ref 8) recommended eutectics EDD/AN for use in shell-loading replacing Amatols

LeRoux (Ref 9) prepd a binary 50/50 eutectic of mp 100° by mixing dry powdered ingredients, followed by heating the ensemble to 105°, with stirring, and then cooling. For the sake of comparison a 50/50-Amatol was prepd by heating TNT to 120° and adding to it, with stirring powdered AN. The EDD/AN was designated as Mixture I, while Amatol was Mixture II: *Detonation Velocity* – 5990m/sec at sp gr 1.62 for I, vs 6150 at 1.60 for II; *Impact Sensitivity* with 10kg wt – 50% detonation at the height of 2m for I & II; *Initiation Sensitivity* – 50g mixt I of sp gr 1.0 required 0.5g MF for deton, vs 0.3g MF for mixt II; *Power* by Coefficient d'Utilisation Pratique (CUP) Method, described in Vol 1, p IX – 116% PA for I, vs 112% for II; *Rifle Bullet Test* at sp gr 0.90 of material packed in boxes: Mixt I detond by bullet of velocity 510m/sec and up, while II detond at 500m/sec and up

Refs: 1) Beil **4**, {484} 2) G.C. Hale, USP 2011578(1935) & CA **29**, 6762(1935)(Prepn & props) 2a) Blatt, OSRD **2014**(1944)(Prepn & props) 3) Allied & Enemy Explosives(1946), p 145(Prepn, props and uses) 4) A.N. Campbell & A.J.R. Campbell, CanJRes **25B**, 96 (1947)(Some props) 4a) ADL PureExplCompds, Pt 2(1947), p 237 5) A.J. Robertson, JSCI **67**, 221–24(1948) & CA **43**, 405(1949)(Thermal decompn of EDAD) 6) H. Ficheroulle, MP **30**, 89–100(1948) (Prepn, props and mixts with AN) & CA **45**, 8249(1951) 7) R.N. Jones & G.D. Thom, CanadJRes **27B**, 830 & 855(1949)(UV Spectra) & CA **44**, 2848(1950) 7a) L. Médard & M. Cessat, MAF **23**, 195(1949) 7b) ADL Synthesis HE's, 1st Report (1949), p 165 8) A. LeRoux, MP **32**, 121–31(1950) & CA **47**, 9014(1953)(Expl props of EDD and of its mixts with AN meltg around 100°, which were found to be suitable for cast-loading as bursting charges of shells in lieu of Amatols) 9) A. LeRoux, MP **34**, 141 & 146(1952)(Properties of 50/50-EDD/AN are listed in comparison with those of 50/50-Amatol) 10) Fedoroff et al, PATR **2510** (1958), pp Ger 35-R, 47-L & 48-L [Forms eutectics: 45/55-EDD/AN and 45/53.5/1.5-EDD/AN/Al, of which the 2nd mixt was used under the name Füller (Filler) No 20 in lieu of Ammonal. The mixture 55/45-EDD/AN, known as Füller 84 was used for cast-loading some shells]

Addnl Refs: A) E.K. Rideal & A.J.B. Robertson, PrRoySoc(Lond) **A195**, 135–50(1948) & CA **43**, 4856(1949)(Calcn of critical hot spot temp) B) L. Médard & M. Thomas, MP **31**, 173–96(1949) & CA **46**, 11684(1952)(Heat of expln) C) Urbański **3**(1965), 470–71

Ethylenediamine-Dinitroform Salt,

$CH_2.NH_2.HC(NO_2)_3$
|
$CH_2.NH_2.HC(NO_2)_3$; mw 362.18, N 30.94%; OB to CO_2 –4.4%, yel crysts, mp 199°(dec), impact sensitivity 28–30cm with 2kg wt. This compd was prepd in the USA after WWII(Ref 3). It is an expl with heat of combstn 1830cal/g at const vol with water liq; and ballistic mortar value 149% TNT. The repts describing its prepn & other props are listed in Ref 2a

It is reported that an aq soln of ethylenediamine (1 mol) & nitroform (2 mols) yielded the yel dinitroform salt, mp 118°(Ref 2a, p 59)

Refs: 1) Beil, not found 2) CA, not found 2a) K. Klager & M.B. Frankel, Aerojet Special Rept **494**(1951), 14, 59 & BIOS/Gr-2 HEC No 5475 3) Dr W.L. Gilliland, Purdue Univ, Lafayette, Indiana 4) ADL 2nd Rept on Synth & Testing of HE's (1951), pp 33, 304, 312, 328, 346 & 374

Ethylenediamine Diperchlorate,

$(CH_2.NH_2)_2.2HClO_4$; mw 281.01, N 9.97%, OB –6.1%; hygroscopic crysts. It can be prepd by reaction of the diamine with Ammonium Perchlorate. Power by Ballistic Mortar 135% TNT; Power by Trauzl Test 144%; Sensitivity to Impact FI > PETN

Refs: 1) Beil **4**, (399) & {484} 2) A.H. Blatt & F.C. Whitmore, OSRD **1085**(1942), 34 3) A.H. Blatt, OSRD **2014**(1944) 4) W.W. Vogl, USP 2406573(1946) & CA **41**,

286(1947)(Addn Compds of Ethylenediperchlorate with acid polynitro aromatic compds) 5) ADL PureExplCompds, Part 1(1947), 69

Ethylenediamine Dipicrate. See 1,2-Bis-(2′,4′,6′-trinitroanilino)-ethane in Vol **2**, p B131-R

Ethylene-N,N′-dinitramine Dipicrate. See 1,2-Bis(2′,4′,6′-trinitronitranilino)-ethane in Vol **2**, p B131-R and Ditetryl or Bitetryl in Vol **5**, p D1513-L

Ethylenediaminetetraacetic Acid (EDTA) $(HOOC.CH_2)_2N.CH_2.CH_2.N(CH_2COOH)_2$. One of the most important *chelating agents* (See under Chelation in Vol 2, p C164-R), mw 292.24; colorless crysts, mp – decomp at 240°; sl sol in w; insol in common org solvents; neutralized by alkali metal hydroxides to form a series of w-sol salts contg from one to four alkali metal cations. It can be prepd by the addn of NaCN and formaldehyde to a basic soln of ethylenediamine to form the tetrasodium salt. Another method is by heating tetrahydroxyethylethylenediamine with Na or K hydroxide, using a CdO catalyst, to form the tetrasodium salt (Ref 4, p 465-R)

It forms numerous salts, some of which are listed in Ref 4, p 466-L. It has been used for determination of about 40 metals and several anions (Ref 2); also used for decontamination of radioactive surfaces and removal of insol deposits of Ca & Mg soaps (Ref 4). Its application in volumetric titrations is discussed in Ref 3. Accdg to Alrose Chem Co, PO Box 1294, Providence, RI, EDTA dissolves Ca oxalate and serves as a *sequestrene*. The term sequestration may be used for any instance in which an ion is prevented from exhibiting its usual props due to close combination with an added material (Ref 4, p 1013-L)

Refs: 1) Beil, not found 2) F Welcher, "The Analytical Uses of Ethylenediaminetetraacetic Acid", VanNostrand, Princeton, NJ(1958) 3) H.A. Flaschka, "EDTA Titrations; an Introduction to Theory and Practice", Pergamon Press, NY(1959) 4) CondChemDict(1961), 465-R & 466-L; and Sequestration, p 1013-L 5) Sax, 3rd edit(1968), 743–44

Ethylenediamine-Trinitromethane. See Ethylenediamine-Dinitroform Salt in this Vol

Ethylenediamine, Mononitro, N-Nitroethylenediamine, or *β-Aminoethylenenitramine,* $H_2N.CH_2.CH_2.NH.NO_2$; mw 105.10, N 39.98%; wh crysts, mp 240°, 244.8°(Ref 2). It was prepd by reaction of $ClCH_2.CH_2.N=C(OPr)-NH.NO_2$ with 10% KOH-ethanol (Ref 2), or by refluxing in water 1-nitro-2-nitriminoimidazolidine (Ref 3)

Refs: 1) Beil, not found 2) R.H. Hall & G.F. Wright, JACS **73**, 2216(1951) & CA **46**, 1989(1952) 3) A.F. McKay et al, JACS **76**, 6371–4(1954) & CA **49**, 15862(1955)

Ethylenediamine, N,N′-Dinitro, ETHYLENEDINITRAMINE, EDNA, Haleite or **1,2-Dinitrodiaminoethane.** (The name Haleite is in recognition of its development as a military explosive by the late Dr G.C. Hale of Picatinny Arsenal), $O_2N.HN–CH_2.CH_2–NH.NO_2$; mw 150.10, N 37.33%, OB to CO_2 –32%; OB to CO –10.5%; wh orthorhombic crysts; sp gr 1.71 (crysts); density – 1.39 for pressed at 10000psi; 1.49 at 20000 and 1.56 at 40000psi; mp 177–79°(starts to decompose at 175°)

Solubilities in 100g of Solvent:

Water: 0.25g at 20°; 0.75 at 40°; 2.13 at 60°; 6.38 at 80° and >20 at 100°

Alcohol: 1.00g at 20°; 2.46 at 40°; 5.19 at 60° and 10.4 at 78°

Ether, chloroform and benzene – very sl sol

It was first prepd in 1888 by Franchimont (Refs 1 & 2), but its value as a military expl was not recognized until Dr Hale prepd and examined it at Picatinny Arsenal (Ref 3)

The earliest method used at PicArsn was in 1933. It consisted of treating ethylenediamine with diethylcarbonate to obtain ethyleneurea, followed by nitration to obtain Dinitroethyleneurea, which on hydrolysis gave Ethylenedinitramine. This process suffered from two disadvantages: the overall yield was only 42% of the theoretical amt; and the initial step, the reaction between ethylenediamine and diethylcarbonate, required the use of high pressures and temperatures over an extended period of time. However, the expl props of EDNA were of sufficient interest to justify further study (Ref 4). See also Aaronson (Ref 5)

With this background, Division 8 of NDRC began work in the fall of 1940 on the synthesis of EDNA that would avoid the unsatisfactory reaction betw ethylenediamine

and diethylcarbonate. Two approaches were investigated: the 1st was a synthesis which did not involve ethyleneurea as an intermediate; the 2nd was an improved synthesis of ethyleneurea (Ref 11)

Laboratory studies developed several methods from which was selected the method of using cheap and available raw materials. When in this synthesis nitration was done with concd nitric acid alone (instead of mixed acid), the overall yield of EDNA was 70% of theoretical and its cost was 30 cents per pound which was considered reasonable

The reactions proceeded as follows:

$$CH_2O + HCN \rightarrow HOCH_2CN \xrightarrow{NH_3} H_2NCH_2CN + H_2O \xrightarrow{2H_2}$$

(98% yield) (82% yield) at high pressure

$$H_2N.CH_2.CH_2.NH_2 \xrightarrow[\text{at } 220^\circ \text{ \& } 820 \text{ atm press}]{CO_2}$$

$$\begin{matrix} CH_2-NH \\ | \\ CH_2-NH \end{matrix}\!\!>CO + H_2O \xrightarrow[98\%\ \text{at RT}]{2HNO_3} \begin{matrix} CH_2-N-NO_2 \\ | \\ CH_2-N-NO_2 \end{matrix}\!\!>CO + 2H_2O$$

$$\xrightarrow[\text{at } 95^\circ]{\text{Hydrolysis}} \begin{matrix} CH_2NHNO_2 \\ | \\ CH_2NHNO_2 \end{matrix}$$

(Ref 11, pp 13 & 14 and Ref 22, pp 153–54)

Ficheroulle (Ref 13, p 89) stated that EDNA can be prepd either by boiling in water Dinitroethyleneurea or by treating Dinitroethylenediurethane with an alcoholic soln of ammonia, followed by acidifying with HCl

$$\begin{matrix} CH_2.N(NO_2)-COOC_2H_5 \\ | \\ CH_2.N(NO_2)-COOC_2H_5 \end{matrix} \xrightarrow{NH_3} \begin{matrix} CH_2(NO_2)-NH_2 \\ | \\ CH_2(NO_2)-NH_2 \end{matrix} \xrightarrow{HCl} \begin{matrix} CH_2.NH.NO_2 \\ | \\ CH_2.NH.NO_2 \end{matrix}$$

Dinitroethylenediurethane

Bachmann et al (Ref 17) investigated possibilities of prepn of EDNA from the following dinitro compds: 1,3-Dinitro-2-imidazolidone; Dinitroethylenebisurethane; N,N'-Dinitroethylenebisacetamide; N,N'-Dinitroethyleneoxamide and 3,3'-Dinitro-1,1'-ethylenebisurea, and he found that EDNA could be obtd from any of these compds with the exception of the 3,3'-Dinitro-1,1'-ethylenebisurea

EDNA is an explosive combining the props of an HE like TNT and an initiating agent like MF or LA. It possesses an advantage of combining a high brisance with comparatively low sensitivity. Usually it is recognized that a HE possessing a very high brisance (eg PETN, RDX or NG) is more sensitive to impact, friction or initiation than the less brisant expls, such as TNT. Another important characteristic of EDNA is its relatively low explosion temp, approaching that of MF or NG. No other HE is known which has such a low sensitivity to impact and at the same time so readily explodes by heat

As EDNA lacks oxygen for complete combustion to CO_2, it may be used in mixts of inexpensive oxidizing agents such as AN, K chlorate, Amm perchlorate, etc, giving a series of expl mixts which are more powerful than EDNA

Properties of EDNA:

Booster Sensitivity – minimum total wt of two Tetryl pellets, which produced 50% detons of EDNA of density 1.42 confined in a cartridge 1-5/8 inches diam by 5 inches long, separated from Tetryl by Acrowax B spacer 1-5/8 inches diam and 2.09 inches thick was 100g (22, p 151)

Brisance by Plate Cutting Test - equivalent to PETN (Ref 7a)

Brisance by Plate Denting Test - 122% TNT for pressed, confined EDNA at 1.50 (22, p 150)

Brisance by Sand Test - 52.3g sand crushed vs 48g for TNT (109%)

Compatibility with Metals (Ref 22, p152)
Unaffected dry: Cu, brass, Al, mild steel, stainless steel, Ni, Cd and Zn
Unaffected wet: stainless steel
Slightly affected dry: Mg & Mg - Al alloy
Heavily corroded wet: Cu, brass, mild steel, Cd, Ni and Zn

Decomposition Equation. The kinetics of decompn of EDNA were studied over the temp range 144.4 to 163.5°C. At the higher temp the sample darkened to a red brn with evolution of NO_2. At lower temp only sl decompn was observed. The true decompn rate is first order. The data followed the Arrhenius equation and are tabulated (Ref 20b)

Decomposition Temperatures: 170° in 20 seconds 173° in 15 secs; 178° in 10 sec; and 189° in 5 secs

Destruction by Chemical Decomposition. EDNA is decompd by addn of hot, dilute

sulfuric acid with evolution of nitrous oxide and ethyleneglycol. It can also be decompd by addition to 5 times wt of 20% Na hydroxide (Ref 22, p 154)

Detonation Velocity - 7570 m/sec for pressed unconfined sample 1.0 inch diam of density 1.49 (Ref 22, p 150); Blatt (Ref 7a) gave 7580 for sample of d 1.50 and 5650 for d 1.0; Ficheroulle (Ref 13) gave 6600 for d 1.25 and 7137 for d 1.50

Explosion Temperature: 0.1 sec (no cap used) 265°; 1 sec 216°(Ref 22, p 150) Blatt (Ref 7a) gave 205° in 1 sec

Exudation - none (Ref 22, p 152)

Flammability Index – 138 seconds. Its definition is given in this Vol, under F's

Fragmentation Test: Sample of 95/5-EDNA/Wax of d 1.56 detonated in 3 inch HE, M42A Projectile produced 600 fragments vs 514 for TNT (Ref 22, p152)

Friction Pendulum Test - unaffected by fiber or steel shoe (Ref 22, p 150)

Gap Test (Fr Transmission de la détonation a l'aire libre)- 14cm distance betn two paper cartridges, each contg 50g EDNA of density 0.80, vs 22cm for Tetryl of d 0.94 (Ref 13)

Gas Volume - 908 cc/g (Ref 22, p 151)

Heat of Combustion - 2477 cal/g (Ref 22, p 151); 369.7 kcal/mole (Ref 7a)

Heat of Explosion - *1276 cal/g (Ref 22, p 151)*

Heat of Formation - 134 cal/g (Ref 22, p151)

Heat Test at 100°(Fr) - litmus paper turns red after ca 48 hours of exposure, vs 3.30 hrs for Ethylenediamine Dinitrate (Ref 13)

Heat Test at 100°(US) - % loss in 1st 48 hrs 0.2, in 2nd 48 hrs 0.3 and no expln in 100 hrs (Ref 22, p 150)

Heat Test at 135° - satisfactory

Hygroscopicity - 0.01% gain at 90% RH (Ref 22, p 150)

Impact Sensitivity, 2 kg Wt - 48cm with BurMines App vs 100cm for TNT, for 20 mg sample or 14 inches with PicArsn App for 17 mg sample (Ref 22, p 150)

Impact Sensitivity (Brit) - FI 61% PA (Ref 7a)

Impact Sensitivity (Fr) (Sensibilité au choc) using 5kg wt app EDNA produced 38 detonations out of 100 trials at the height of 1.50 meters, vs 70 detons for Ethylenediamime Dinitrate and 95 detons for Tetryl (Ref 13)

Initiation Sensitivity to - minimum detonating charge 0.21g MF or 0.13g LA (Ref 22, p 150); 0.20g MF for EDNA at d 0.8 (Ref 13)

International Heat Test at 75° - % loss in 48 hrs - 0.01 (Ref 22, p 150)

Loading Densities - 1.49, when pressed at 20000 psi; 1.28 at 5000; 1.38 at 10000; 1.41 at 12000 and 1.44 at 15000 (Ref 22, p 152)

Power by Ballistic Mortar - 136% TNT (Ref 7a); 139% TNT (Ref 22, p 150)

Power by Trauzl Test - 118% PA (Ref 7a); 122% TNT (Ref 22, p 150)

Power by Modifidd Trauzl Test [Fr Coefficient d'Utilisation Pratique (CUP), described in Vol 1, p IX]- *129% PA* (Ref 13)

Rifle Bullet Impact Test - Similar to TNT, but less sensitive than RDX (Ref 7a); in 100 trials there were 60 partial detons, 20 burnings and 20 unaffected (Ref 22,p150)

Sand Test. See under Brisance by Sand Test

Sensitivity to Impact. See Impact Sensitivity

Sensitivity to Initiation. See Initiation Sensitivity to

Stability at 135°. See Heat Test at 135°

Storage - dry (Ref 22, p 152)

Surveyance - slight lowering of mp, but no increase in stability after storage for 4 months at 65° (Ref 7a)

Thermal Stability at 135°. See Heat Test at 135°

Uses. See below

UV Spectra. See Ref 15

Vacuum Stability tests: at 100° - 0.5 cc/5g in 48 hrs; at 120° - 1.5cc and 135° 11+cc (Ref 22, p 150); at 120° - 2.4cc/5g in 48hrs (Ref 7a)

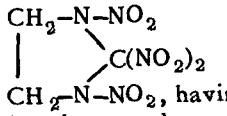
Velocity of Detonation - See Detonation Velocity

Volatility - nil (Ref 22, p 15)

Uses: It was proposed as a pressloaded charge in booster both in USA (Ref 22,p 152) and in France by Ficheroulle (Ref 13). He also proposed a mixture of EDNA 38.5 & AN 61.5%, which he designated as **NB77**; it was more powerful than EDNA, sl less stable, had gap test value 11cm and deton velocity 6800m/sec at d 1.60

Refs for EDNA: 1)Beil **4**, 572 & (569) 2) A.P.L. Franchimont & E.A.Klobbie, RecTrav Chim **7**, 17 & 244 (1888) 3)A.P.L. Franchimont, Rec **29**, 308 (1910) 4)G.C.Hale, USP 2011578 (1935) & CA **29**, 6762 (1935) 5) H.A. Aaronson, USP 2149260(1939) & CA **33**, 4273 (1939) 6) W.E. Bachmann, OSRD **819** & PBA **31199** (1942) (EDNA and related

compds) 6a) Expls Res Lab, OSRD **1734**, (1943) (Stability of EDNA and related nitramines) 7) Davis (1943) 393-94 (Ethylenedinitramine and its Ag & K salts) 7a) A.H. Blatt, OSRD **2014** (1944) (Compilation of data on organic explosives) 9) E.I.duPont de Nemours & Co. Inc., OSRD **3466** (1944) (Production of Haleite from ethyleneurea) 10)A.T. Blomquist & F.T. Fiedorek, OSRD **4134** or PB **18867** (1944), pp 6-15 (Aliphatic nitramines, which include EDNA and its salts) 10a) Anon, "Allied and Enemy Explosives" (1946), p143 (Props of EDNA) 11) Coll, "Summary Technical Report of Division 8, NDRC", Vol 1, Washington, DC (1946) 12-14(Prepn of EDNA) 11a) G.V.Caesar & M. Goldfrank, USP 2400288 (1946) & CA **40**, 4526 (1946) (Prepn) 11b) A.M. Soldate & R.M.Noyes, AnalChem **19**, 442-4 (1947) & CA **41**, 6105(1947)(X-ray data) 12) A.J.B. Robertson, Trans Farad Soc **44**, 677 (1948) (Thermal decompn of EDNA) & CA **43**, **2775** (1949) 13) H. Ficheroulle, MP **30**, 89-100 (1948) & CA **45**,8249 (1951) (Prepn & props of EDNA and its ammonium salt. Binary mixts of these compds with AN) 13a) ADL Synthesis HEs, 1st Rept (1949) (Some props of EDNA) 14) G.C.Hale; USP 2472105 (1949) & CA **43**, 6827 (1949) (Expls which may be cast at temp of low pressure steam in shells, bombs, etc consist of suspension of EDNA > 40% in molten TNT) 15) R.N. Jones & G.D.Thorn, CanadJRes **27B**, 829 (1949) (UV spectra) & CA **44**, 2848(1950) 16) M. Carnack & J.J. Leavitt, JACS **71**, 1221(1949) & CA **43**, 5307(1949) (UV spectra) 17) W.E. Bachmann, JACS **72**, 3132–38(1950)(Possibilities of prepn of EDNA from some dinitro compds) & CA **44**, 10656(1950) 17a) ADL, Synthesis HE's, 2nd Rept (1951), p 223 (It was suggested by Dr C.R. Morgan that condensation of Dibromodinitromethane with EDNA salts should lead to the cyclic structure,



CH_2–N–NO_2, having zero oxygen balance, but the compd was not prepd); pp 258 & 287, ADL PureExplCompds, Part 2(1947)(Heats of combustion, explosion and formation of EDNA) 18) H. Henkin & R. McGill, IEC **44**, 1391–95(1952) & CA **46**, 8858(1952)(The lowest expln temp as observed for 25mg sample of EDNA, when tested in modified Marshall apparatus was about 175° for 5 secs; for 4.8 secs it was 180°) 19) W.R. Tomlinson, Jr, JOrgChem **17**, 648–68(1952) & CA **47**, 2125(1953)(Decompn of Haleite under reduced pressure in the presence of water, glycol or TNT) 20) M.A. Cook et al, JPhysChem **58**, 1114–24(1954) & CA **49**, 2801(1955) [Equation of state of EDNA: $D^2 = nRT_2e^x(1+\theta)^2/\theta$] 20a) C.P. Spaeth, USP 2684384(1954) & CA **49**, 6993(1955)(Prepn) 20b) M.A. Cook & M.T. Abegg, IEC **48**, 1092–93(1956) 21) L. Médard & M. Thomas, MP **37**, 129 & 138(1955) & CA **51**, 716(1957)(Heat of combstn at const vol) 21a) Anon, TM9 **1300–214**, "Military Explosives", (Nov 1967), pp 7–58 to 7–61 22) P. Tavernier, MP **38**, 309 & 329(1956) & CA **51**, 15952(1957)(Heat of formation at C_v 148cal/g and at C_p 176) 22a) W.R. Tomlinson, Jr & O.E. Sheffield, "Properties of Explosives of Military Interest", PATR **1740**, reissued in 1971 as Army Materiel Command Pamphlet **AMCP 706–177**, pp 150–55 (Haleite)

Addnl Refs for EDNA:

A) Thorpe **4**(1940), 489(Prepn & props of EDNA)

B) J.D. Hopper, PATR **1113**(1941) & **1162**(1942) (Study of manuf of EDNA on a semiplant scale)

C) H.A. Aaronson, PATR **1198**(1942)(Stability of EDNA and its mixts with TNT)

D) H.A. Aaronson, PATR **1199**(1942)(Development of assay method for EDNA)

E) H.A. Aaronson, PATR **1200**(1942)(Development of assay method for EDNA)

F) H.A. Aaronson, PATR **1232**(1943)(Development of assay method for Ethyleneurea)

G) W.R. Tomlinson, PATR **1252**(1943)(Study of rates of hydrolysis of Dinitroethyleneurea &EDNA

H) A.L. Sandifer, PATR **1279**(1943)(Study of manuf of EDNA on a semi-plant scale)

I) H.A. Aaronson, PATR **1288**(1943)(Study of manuf of EDNA on a semi-plant scale)

J) H.A. Aaronson, PATR **1294**(1943)(Stability of EDNA and its mixts with TNT)

K) J. Rubin, PATR **1319**(1943)(Manuf of EDNA on a semi-plant scale)

L) Clift & Fedoroff, Vol 1(1943), p E3 (Ethylenedinitramine or N,N′-Dinitroethylenediamine)

M) J. Rubin, PATR **1378**(1944)(Props of Ammonium Salt of Haleite)

N) H Aaronson, PATR **1395**(1944)(Stability

of EDNA and its mixts with TNT)
O) D.R. Cameron, PATR **1400**(1944)(Fragmentation tests of 37mm M36 and 105mm M38A1 Shell loaded with Haleite, Ednatol and other explosives)
P) S. Sage, PATR **1434**(1944)(Properties and military characteristics of Haleite & Ednatol)
Q) H. Aaronson, PATR **1600**(1946)(Preparation & props of nitrated condensation product of Haleite, formaldehyde and ammonia)
R) F. Pristera, PATR **1796**(1951)(A method for staining Cyclotols, Ednatols, Pentolites and Picratols for observing and studying non-TNT phase)
S) S.M. Kaye, PATR **1937**(1953)(Determination of purity of EDNA by titration in nonaqueous medium)

Ethylenedinitramine (EDNA or Haleite). Specification Requirements and Tests

Applicable Specification: PXS–1033(Rev 2), 24 July 1943 and Amend 1, 7 April 1947

Specification Requirements:

1. Moisture – 0.10% (max)
2. Color – white to buff
3. Melting Point – 174°(min)
4. Purity – 99.0% (min)
5. Insoluble Matter – 0.10% (max)
6. Granulation:
   Thru US Std Sieve No 10 – 100%(min)
   Thru US Std Sieve No 100 – 20% (max)
7. Grit – none

Laboratory Tests:

1. Moisture. Transfer a portion of approx 5g of the sample to a tared weighing dish of at least 1.5 inches diameter, and weigh. Place the dish in a vacuum desiccator containing sulfuric acid and evacuate to a pressure not greater than 150mm of Hg. After 30 mins, remove the dish, and re-weigh. Calculate any loss in wt to percentage of moisture in the sample

2. Color. Determine by visual examination

3. Melting Point

A. Apparatus. Set up a melting point bath equipped with a mechanical stirrer and a source of heat that can be easily regulated. A beaker of 1 to 2 liters capacity about ¾ full of clear peanut oil is recommended. Suspend an accurately standardized total immersion Centigrade thermometer in the bath so that the bulb is not less than 1.5 inches from the bottom of the bath. If the mercury column will not be completely immersed at the temperature of the observed melting point, suspend a second thermometer about 0.5 inch from, and with its bulb at about the height of the middle of the exposed mercury column of the first thermometer

B. Determination. Use thin-walled capillary tubes of uniform diameter, long enough to extend beyond the top of the bath. Fill the tube with dry sample to a depth of approx 4mm, and compact the material by tapping. Start the stirrer and heat the bath rapidly to 165°C, and then at such a rate that the rise in temperature is 1.0°±0.5°C in 2 minutes. When the temperature reaches 172°C, attach the melting point tube so that its lower end is in contact with the bulb of the thermometer. Continue beating so that the temperature continues to rise at the same rate. Consider the uncorrected melting point to be the temperature at which the solid in the tube softens and breaks up into sections which usually rise in the tube

C. Correction. If the mercury column is completely immersed at the above temperature, report this temperature, with calibration corrections applied, as the melting point of the sample. If part of the column is exposed, add the following correction to the above temperature:

Correction = n (T–t) x 0.000159

Where:

n = Number of degrees in the exposed column
T = Uncorrected melting point
t = Average temperature of exposed mercury column

4. Purity. Transfer from 1.1 to 1.2 grams of the sample to a tared weighing dish and weigh accurately. Transfer the weighed portion to a clean 500cc Erlenmeyer flask by means of distilled water and wash down the neck and walls. Use a total of 100 to 125cc of distilled water. Add 48 to 49cc of N/3 sodium hydroxide solution with constant swirling of the contents of the flask so as to hasten solution of the Haleite. Heat the flask on a hot plate until the contents are boiling and by means of distilled water, wash down any undissolved material adhering to the wall of the flask. When solution is complete, cool the flask and contents to room temperature. Add 10 drops of phenolphthalein solution (0.5g per 100cc of alcohol) and titrate with approx N/5 sulfuric acid solution until the solution is a faint pink or has become colorless. In the latter case,

titrate with a N/3 sodium hydroxide until a pink color reappears. Correct the volumes of alkali and acid to standard temperature, and calculate the purity of the sample

$$\text{Percentage purity} = \frac{7.505\ (AB-CD)}{E}$$

Where:

A = cc of sodium hydroxide solution used
B = Normality of sodium hydroxide solution
C = cc of sulfuric acid solution used
D = Normality of sulfuric acid used
E = Weight of sample used

5. *Insoluble Matter.* Transfer a weighed portion of approximately 10g of the sample to a 250cc beaker. Add 200cc of distilled water and heat to boiling. When solution is complete, filter through a tared Gooch crucible. Wash the beaker and insoluble residue in the crucible thoroughly with boiling distilled water until free from soluble material. Dry the crucible and contents at 100°C for 2 hours, cool in a desiccator, and weigh. Calculate the weight of residue in the crucible to percentage of insoluble matter in the sample

6. *Granulation.* Transfer a 100g portion of the sample to a No 10 US Std sieve superimposed on a No 100 US Std sieve and equipped with a bottom pan and cover. Shake the assembly for three minutes. Weigh any material retained on the sieves and calculate the percentage of the sample passing thru each sieve

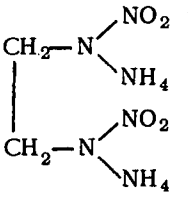
7. *Grit.* Transfer a 25g portion of the sample to a 1 liter beaker and add 500cc of boiling distilled water. Heat until solution is complete. Pour the solution thru a No 60 US Std sieve and wash any residue on the sieve with a jet of hot distilled water. When no more insoluble material passes thru the sieve, dry the sieve and residue remaining on it. Transfer the dry residue to a piece of paper and then to a smooth glass slide. Rub the material on the glass by exerting pressure with a smooth steel spatula blade. Note if particles of grit are present as indicated by lack of uniformity of the material and the persistence of a scratching noise when pressing and rubbing of the material on the glass is continued

Ref: A.R. Lusardi, "A Manual of Laboratory Procedures for the Analysis and Testing of Explosives and Pyrotechnics", Method No 107, Expls & Proplnts Lab, FeltmanResLabs, Picatinny Arsenal, Dover, NJ (1962)

EDNA SALTS

Ethylenediamine-N,N′-dinitro Salts with Ammonium and Metals:

Diammonium Ethylenedinitramine, (AEDNA)



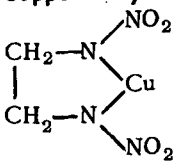
mw 184.17, N 45.64%, OB −52.2%; crysts, sp gr 1.505 at 18°; mp 158°(Ref 9), decomp at 180°(Ref 6); sol in w; insol in absol alc or in eth. It can be prepd by saturating ethylenedinitramine with ammonia, separating the crysts and purifying them; for this they are dissolved in 20% ammonia and pptd by ammoniacal abs alc, followed by pressing the crysts on a fritted glass, rinsing with ammoniacal abs alc and drying in cold over KOH. (Drying over H_2SO_4 or $CaCl_2$ results in loss of some NH_3) (Ref 9). B & F (Ref 3) prepd it by warming EDNA in alc with concd ammonium hydroxide

Following are its other props (Refs 3, 5, 6 & 9)

Brisance – no info; *Decomposition Temperature* – 180° with evolution of white fumes; *Detonation Velocity* – 7132m/sec at density 1.35; *Heat of Combustion* at C_V and 18° – 519kcal/mole; *Heat of Formation* at C_V – 74.6kcal/mole; *Hygroscopicity* – very high; *Impact Sensitivity with 5kg Weight* – at the heights 1.0 to 1.5 meters only 2 detonations out of 100 drops; *Initiation Sensitivity* – detonates very difficultly at density 0.70 by means of 3g MF; *Power by CUP Method* (Modified Trauzl Test) – 69.5% PA vs Tetryl 120%; *Stability* – low

Uses: In the opinion of Ficheroulle (Ref 6), AEDNA could be useful when an expl with extremely low sensitivity is required. He proposed the following binary mixture **NB78–** AEDNA 27.7 & AN 72.3% with favorable OB to CO_2 and found that although its power (by CUP) is high (104.4% PA), its sensitivity to impact is too high for use as a bursting charge in shells in lieu of *Schneiderite* – (DNN 87–88 & AN 13–12%); and besides it is more expensive than Schneiderite

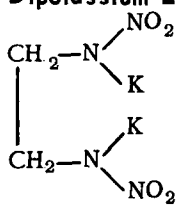
Copper Ethylenedinitramine,



mw 211.62, N 13.24% (NO_2), OB −22.6%

Accdg to Blatt (Ref 4) its impact sensitivity is 17cm for 50% explns using a 5kg wt; its FI is 30–40% PA and it ignites w/o deton at 350°

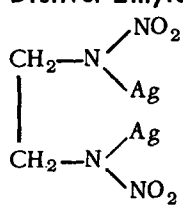
Dipotassium Ethylenedinitramine,



mw 226.18, N 12.38% (NO_2), OB −21.2%, ndls (from alc). Accdg to Ref 7, the mono-K salt is amorphous, and the di-K salt is cryst but is a dihydrate when prepd from an aq system. The di-K salt was prepd by cooling a warmed soln of KOH, water, 95% ethanol & EDNA

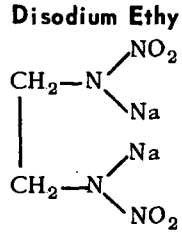
Blatt (Ref 4) gives its impact sensitivity as 43cm vs 51cm for RDX with 5kg wt for 50% explns

Disilver Ethylenedinitramine,



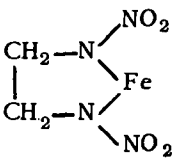
mw 363.82, N 7.70% (NO_2), OB −13.2%, wh powd. It is prepd by adding a soln of the K salt to excess $AgNO_3$ (Ref 1)

Disodium Ethylenedinitramine,



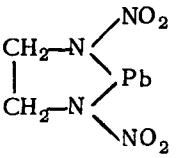
mw 194.06, N 14.43% (NO_2), OB −24.7%. Accdg to Ref 7, the mono-Na salt is amorphous, and the di-Na salt cryst in ndls (from water); prepd by dissolving EDNA in 10% NaOH and pptg w alc

Iron Ethylenedinitramine



mw 203.93, N 27.47%, crysts, insol in water. Its impact sensitivity is 21cm for 50% explns using 5kg wt vs 51cm for RDX in same test. It is formed by adding Pb nitrate to an aq soln of the Na compd (Ref 4)

Lead Ethylenedinitramine,



mw 355.27, N 7.88% (NO_2), OB −18.0%. Accdg to Ref 8, the Pb salt is formed by dissolving equimolar parts of EDNA in sodium hydroxide solution and then adding an equimolar part of lead nitrate solution and filtering the pptd lead salt. The salt is heat stable up to 300°C and crushes 17g of sand in a sand bomb

Blatt (Ref 4) lists its impact sensitivity as 30cm for 50% explns using a 5kg wt vs 51cm for RDX in same test; its FI is 49% PA and ignites at 310° in 1 sec

Refs for EDNA Salts: 1) Beil **4**, 573 (K and Ag) 2) G.C. Hale, USP 2011578 & CA **29**, 6762 (1935) (Some salts) 2a) Davis (1943), 394 (Ag and K salts of EDNA are mentioned) 3) A.T. Blomquist & F.T. Fiedorek, OSRD **4134** & PB **18867** (1944), 82 (Dipotassium salt); 83 (Diammonium salt) 4) Blatt, OSRD **2014** (1944) (Sensitivities to impact of K, Fe, Pb and Cu salts) 5) J. Rubin, OSRD **1378** (1944) (Ammonium salt) 6) H. Ficheroulle, MP **30**, 89–100 (1948) & CA **45**, 8249 (1951) (Prepn and props of ammonium salt and its mixts with AN) 7) N. Allentoff & G.F. Wright, ActaCryst **6**, 1–8 (1953) & CA **47**, 6733 (1953) (K and Na salts) 8) A.O. Franz, USP 2708623 (1951 & CA **49**, 14326 (1955) (Pb salt for use in expl compns) 9) L. Médard & M. Thomas, MP **38**, 46, 53 & 63 (1956) & CA **51**, 13553 (1957) (Prepn and props of diammonium salt) 10) Anon, TM **9-1300-214**, "Military Explosives" (Nov 1967), p 7–59

EDNAFIVE (Ethylenedinitramine–Fivonite) 50/50 Ednafive is a castable HE composed of EDNA 50 & Fivonite (Tetramethylolcyclopentanone Tetranitrate) 50 + 1% catechol. Its power is 130% TNT by Ballistic Mortar Test and brisance by PDT (Plate Dent Test) is 111% of TNT. In the Bullet Impact Test of 15 1-inch pipe nipples, 9 samples were not affected and 6 partially exploded. A 60/40 Ednafive mixt in the same latter test of 13 nipples showed 5 samples not affected, 7 exploding and 1 detonating high order; in a welded can the test of 5 samples showed 5 partially exploding

Fivonite alone has a PDT value of 106–112%, BMT 121–122% and Trauzl Test value 129% of TNT. Its vel of detonation is 7300m/sec at sp gr 1.57

Ref: E.H. Eyster & W.H. Rogers, "Physical Testing of Explosives", OSRD **5745** (Nov 1945)

EDNATOLS. Combinations of EDNA (Ethylenedinitramine) and Tol (TNT) in various proportions. The most used mixts are EDNA/TNT–60/40, 55/45 and 50/50. They are powerful expls which melt at ca 80°C and can be cast at ca 85°C, giving yellowish uniform blends suitable as bursting charges in ammunition. These expl mixtures were developed at Picatinny Arsenal just prior to WWII. For their prepn wet EDNA was added slowly to molten TNT heated to ca 105°C in a steam jacketed melting kettle equipped with a stirrer. Heating and stirring were continued until all moisture was evaporated. Loading in ammunition was done by pouring the mixture cooled to 85°

Although the properties of mixts 60/40, 55/45 and 50/50 are known (as detd at PicArsn), it is sufficient to list here only the props of 55/45–Ednatol, as was done by Tomlinson & Sheffield (See Ref 2)

Properties of 55/45–EDNA/TNT Mixture

Oxygen Balance to CO_2 –51% and –17% to CO

Density (cast) – 1.62g/cc

Blast (Relative to TNT) in Air: Peak Pressure 108%, Impulse 110% and Energy 108%

Blast Under Water: Energy 113%

Booster Sensitivity Test (See Vol 1 of Encycl, p VIII), using 100g Tetryl pellet. Thickness of Acrawax B pellet to achieve 50% detonations was 1.28 inches

Brisance by Sand Test in 200g Bomb – 112% TNT

Brisance by Plate Test B – 112% TNT

Eutectic Temperature – ca 80°

Explosion Temperature in 5 secs – ca 190°(dec)
in 1 sec – ca 248°(dec)

Exudation Test at 65° – no exudation

Flammability Index – Will not continue to burn

Fragmentation Test – in 3-inch M42 Shell – 118% TNT

Fragment Volocity – 2730ft/sec at 9 ft and 2430 at 25.5 ft

Friction Pendulum Test – unaffected by fiber or steel shoe

Heat Test at 100°C – % Loss in 1st 48 hrs 0.2
% Loss in 2nd 48 hrs 0.1
No expln in 100 hrs

Hygroscopicity, % Gain at 30° & 90% RH – none

Impact Sensitivity, BurMinesApp, 2kg wt, 95cm for 20mg sample

Initiation Sensitivity – Minimum detonating charge: MF 0.22–0.23g. Did not deton consistently with No 8 DuPont cap

Metal Compatibility:

When dry: Brass, Al, steels, Cd & Ni are unaffected, while Cu, Mg, Al-Mg alloy are slightly affected

When wet: Stainless steels are unaffected; Al sl affected; Cu, brass, Mg, Mg-Al alloy, mild steels are considerably affected

Power by Ballistic Mortar Test – 119% TNT

Power by Trauzl Test – 120% TNT

Rifle Bullet Impact Test – Burned 7%, Unaffected 93%

Shape Charge Effectiveness – ca 120% of TNT

Storage – Dry; Hazard Class 9, Compatibility Group I

Vacuum Stability at 100° – 1.0cc gas evolved in 48 hrs

Vacuum Stability at 120° – 11+cc

Velocity of Detonation – 7340m/sec for 1 inch diam cast unconfined chges. Used in shells, bombs and special ammunition components as an alternate explosive

Refs: 1) Anon, Allied and Enemy Explosives (1946), p 144 2) W.R. Tomlinson, Jr and O.E. Sheffield, PATR **1740**, Revision 1(1958). Reproduced in 1971 as Pamphlet **AMCP 706–177** by the US Army Materiel Command, Washington, DC 20315 (Included 8 refs and a list of PATR's discussing prepn, props and tests for Ednatols)

Ednatol, Aluminized. A mixture of EDNA 40, TNT 20 & Al 20, OB to CO_2 –60.2%. This

castable HE was found to have a calcd heat of expln of 142kcal/mol, temp developed on expln 5056°K, and power by Ballistic Mortar 130% of TNT

Ref: ADL PureExplsCompds, Part 3 (May 1950), p 380 and Part 4 (May 1952), p 589

Ethylenediamine-N,N′-Dinitro-Glycolicdiurethane Dinitrate or **N,N′-Dinitro-N,N′-bis-(2-nitratoethoxycarbonyl)-ethylenediamine** (CA). (Called Dinitrate de diuréthane glycolique de l'éthylène-dinitramine, in French),

$CH_2.N(NO_2).COO.CH_2.CH_2.NO_3$
|
$CH_2.N(NO_2).COO.CH_2.CH_2.NO_3$; mw 416.23, N 20.02%; OB −30.7%; crysts, mp 85.0–85.1° (Desseigne gave 84.5°). It can be prepd by nitrating N,N′-bis(2-hydroxyethoxycarbonyl)-ethylenediamine with Ac_2O & HNO_3 below 5° (Ref 4). It decompd exothermally on reaction with pyridine, forming water-insol products. Heat of combstn at C_V and 18°931.4kcal/mole, and heat of formation 229.6kcal/mole (Ref 3). This expl compd is nonhygroscopic and does not hydrolyze in humid air nor in water below its mp (Ref 2)

Refs: 1) Beil, not found 2) C. Desseigne, FrP 1094959 (1955) & CA **53**, 1159 (1959) 3) L. Médard & M. Thomas, MP **38**, 47, 54 & 63 (1956) & CA **51**, 13553 (1957) 4) E.S. Lane, JChemSoc **1956**, 2006 & CA **51**, 264 (1957) (Prepn)

Ethylenedibromide, Ethylenebromide or **1,2-Dibromoethane**, $BrCH_2.CH_2Br$; mw 187.88; colorless, nonflammable, poisonous liquid; sp gr 2.180 at 20°/4°, fr p 10°, bp 131.7°, n_D 1.5357 at 25°; sl sol in w; miscible with most solvents and thinners. It can be prepd by the action of bromine on ethylene gas; used as scavenger for lead in gasoline; as solvent for fats, oils, waxes, gums, etc and in prepn of celluloid. Avoid breathing vapor; absorbs thru skin

Refs: 1) Beil **1**, 90, (28) & [61] 2) Cond-ChemDict (1961), 466-L 3) Sax, 3rd edit (1968), p 744

Ethylenedicarboxylic Acid. See Succinic Acid

Ethylenedichloride. See 1,2-Dichloroethane in Vol 5, p D1209-L and Sax, 3rd edit (1968), p 744

Ethylenediglycolamide and Derivatives

N,N′-Ethylenediglycolamide, $HO.CH_2.CO.NH.CH_2.CH_2.NH.CO.CH_2OH$; mw 167.17; crysts, mp 141°. It can be prepd by slowly adding 17.5g of 68.8% aq ethylenediamine to 36g of methyl glycolate. After addn, the soln is partly evaporated by heating under reduced pressure on a water bath and then cooled. The resulting crysts are separated and purified by recrystallization from 95% ethanol (Ref 2)

It gives on nitration the following expl compds:

N,N′-Ethylenediglycolamide Dinitrate, $O_2NO.CH_2.CO.NH.CH_2.CH_2.NH.CO.CH_2.ONO_2$; mw 266.17, N 21.05%; OB −54.1%, wh crysts, mp 161–62°. It can be prepd by dissolving 10g of ethylenediglycolamide in 50ml of wh nitric acid, followed by heating the soln for 15 mins at 50–60°. On pouring the soln into large vol of ice-water, a wh solid separates which can be filtered off, washed with w, dried, recrystallized from chlf contg a small amt of alc (Ref 2). No props are given in Ref 2

Nitro-N,N′-ethylenediglycolamide Dinitrate, $O_2NO.CH_2.CO.N(NO_2).CH_2.CH_2.NH.CO.CH_2.ONO_2$; mw 311.17, N 22.51%, OB to CO_2 −33.4%; crysts, mp 100–101°(decomp). It can be prepd by dissolving 2g of ethylenediglycolamide in a cold mixture of 5ml of wh nitric acid and 8ml of acetic anhydride, allowing to stand for 10 mins at RT and then pouring the soln on ice. The resulting wh solid is collected on a filter, washed with w, dried and crystallized from chlf contg some alc (Ref 2). No props are given in Ref 2

Dinitro-N,N′-ethylenediglycolamide Dinitrate, $O_2NO.CH_2.CO.N(NO_2).CH_2.CH_2.N(NO_2).CO.CH_2.ONO_2$; mw 356.17, N 23.60%, OB to CO_2 −18.0%; wh crysts, mp 135–36°. It can be prepd by dissolving 2g of Nitro-N,N′-ethylenediglycolamide Dinitrate in 8ml wh nitric acid, heating the soln at 60° for 30 mins and pouring on ice. Then wh solid is separated by

filtration, washed with w, dried and crystallized from a mixture of chlf & ethyl acetate (Ref 2). No props are given in Ref 2

Refs: 1) Beil, not found 2) R. Adams & C.S. Marvel, OSRD **86**(1941), pp 12–13, 37–42

Ethylenedinitrilotetraacetic Acid. Same as Ethylenediaminotetraacetic Acid

2,2′-(Ethylenedinitroimino)-diethanol Dinitrate. See 2,2′-(Ethylenebisnitroimino)-diethanol Dinitrate under 2,2′-(Ethylenebisimino)-diethanol and Derivatives in this Vol

1,5-Ethylene-3,7-dinitro-1,3,5,7-tetraaza-2,4,6,8-cycloöctane. See 3,7-Dinitro-1,5-endoethylene-1,3,5,7-tetraza-2,4,6,8-cycloöctane under 1,5-Endoethylene-1,3,5,7-tetraza-2,4,6,8-cycloöctane and Derivatives in Vol **5** of Encycl, p E90-L

Ethylenedioxyamine (called *a,a′*-Äthylendihydroxyamin in Ger),

$CH_2.O.NH_2$
|
$CH_2.O.NH_2$; mw 92.10, N 30.42%, sl aromatic liq, bp 203° & 105° at 23mm press, explodes mildly under distillation at normal press; miscible with water, alc & chlf. It was prepd from the K salt of 1,1′-ethylenedioxyamine-2,2-disulfonic acid by reaction with perchloric acid and heating the aq soln with concd alkali added

The following salts were prepd:

Hydrochloride Salt, $C_2H_8N_2O_2 + 2HCl$; shiny pltlts, mp 233°

Nitrate, $C_2H_8N_2O_2 + 2HNO_3$, mp 124°, detonates on heating above its mp

Sulfate, $C_2H_8N_2O_2 + H_2SO_4$, crysts (from w), mp 260°; v sl sol in alc, acid in aq soln

Refs: 1) Beil **1**, 470 & [523] 2) W. Traube et al, Ber **53**, 1489(1920) & CA **15**, 84(1921)

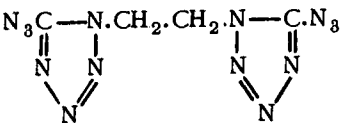
Ethylenedioxyamine Perchlorate (EDAP) $H_2NOCH_2.CH_2.ONH_2 + 2HClO_4$. Accdg to Rept EI-221, Dept of Defense, Armed Services Explosives Safety Board, Washington, DC 20315 as quoted in Ref: "A low-order explosion of EDAP is believed to have occurred while it was being purified. A chemist had been working on the synthesis and characterization of this sensitive material. Both men present in the room died. Explosion caused fire, a minor secondary explosion, noxious irritating fumes and smoke, damage to contents of the lab and an adjacent lab"

Ref: Expls & Pyrots, The Franklin Inst Newsletter Vol **1**, No 6 (June 1968)

Ethylenedioxybenzene or Pyrocatechol, Ethylene Ester. See Benzodioxan in Vol 2, p B66-R

Ethylenediphenyldiamine. See N,N′-Diphenylethylenediamine in Vol 5, p D1457-R

Ethylenditetrazylazide,



mw 248.18, N 79.04%. It is a highly brisant compd which can be easily initiated by sparks or flame. When absorbed in a porous combustible material like cellulose or paper, it has still an excellent igniting power for expls. It gelatinizes NC, thus forming an easily ignited gelatin of high brisance

Friederich patented the compd for use in primers and detonators and as a core of detonating fuses

Refs: 1) Beil, not found 2) W. Friederich, USP 2170943(1939) & CA **34**, 265(1940)

1,1′-Ethylenediurea Dinitrate. See under 1,1′-Ethylenebisurea and Derivatives in this Vol

Ethylenediurethane Azide. See under Ethylenebisurethane Azide and Derivatives in this Vol

ETHYLENEGLYCOL AND DERIVATIVES

Ethyleneglycol, Ethanediol-1,2, Glycol (Abbr by us Gc); Ger names are Glykol or Äthylenglykol; $HOCH_2.CH_2OH$; mw 62.07; colorless,

syrupy, sweet-tasting liquid; sp gr 1.1155 at 20°; d(true) 1.1136 at 20°; fr p −13.5°, bp 197.2°, n_D 1.4316 at 20° & 1.430 at 25°; flash p (Cleveland open cup) 240.8°F, fire p 250°F. Solubilities and other properties of Gc are listed after the prepn

Preparation of Ethyleneglycol

It was first prepd in 1856 in France by C.A. Wurtz (pronounced Würtz) (1817–1884) by saponification of ethyleneglycoldiacetate with KOH, and its structure was established in 1859. In 1860 it was prepd by the hydration of ethyleneoxide (Refs 1 & 7a). For about a half century Gc remained a laboratory curiosity. Accdg to Naoúm (Ref 3, p 211), the first statements for using it in prepn of EGDN (Ethyleneglycol Dinitrate) proposed as a possible additive to NG (Nitroglycerin) for lowering its fr p – appeared in GerP 179789 of 1904, EnglP 12770 (1912) and in MP **16**, 73(1912) & **17**, 175(1914). However, at that time there appeared to have been no commercial manuf of Gc or its dinitrate. The scarcity of glycerin in Germany during WWI turned attention to NGc as a possible substitute for NG, and all methods for prepg it were studied. The commercial process of manufg Gc from ethanol via ethylene, ethylenechlorohydrin and ethylenedichloride was therefore developed, and during WWI many thousands of kilos were manufd, mainly by the firm of Th. Goldschmidt in Essen, and furnished the explosives industry. It was nitrated to NGc, partly as such and partly in mixts with NG as a completely equivalent substitute for the latter in mining expls (Ref 3)

Accdg to Curme & Johnston (Ref 15), manuf in the USA of Gc and NGc began in 1925, when the first large scale plant was established by Carbide and Carbon Chemical Corp at South Charlston in West Virginia. In 1927 the USBurMines approved 25 ethyleneglycol derived expls as being permissible for use in coal mines and by 1929, practically every manufacturer of Dynamite in the USA was using EGDN as an antifreeze component (See also under DYNAMITE in Vol 5, pp D1590 to D1593-R, D1594-L, D1599 & D1601

Manufacture of Ethyleneglycol. Several methods of manuf, mostly of German origin, are described in Naoúm (Ref 3, pp 212–16)

Most US plants used at the time of publication of Curme & Johnston's book (Ref 15) are of the following three methods:

1) Hydration of ethyleneoxide, which can be prepd from ethylene by direct action of oxygen in presence of a catalyst (such as silver). This process is based on Lefort's discovery of 1931

$$H_2C{:}CH_2 + 0.5O_2 \xrightarrow{Ag} H_2C.O.CH_2 + CO_2 + H_2O$$

$$H_2C.O.CH_2 + H_2O \rightarrow HOCH_2.CH_2OH$$ (Yield ca 50%) (See also Ref 13)

2) Hypochlorination of ethylene to ethylenechlorohydrin, followed by hydrolysis with Na bicarbonate:

$$H_2C{:}CH_2 + HOCl \rightarrow HO.CH_2.CH_2Cl$$

$$NaHCO_3 + H_2O \rightarrow HOCH_2.CH_2OH + CO_2 + H_2O + NaCl$$

The yield is 65–70%

Note: Ethylene can be prepd in high yield by passing ethanol vapor over coke wet with phosphoric acid in towers (Ref 3, p 212, footnote 11)

3) Formaldehyde is made to react at high pressure and high temperature with CO and water to produce glycolic acid:

$$CH_2O + CO + H_2O \rightarrow HOCH_2.COOH$$

The acid is purified by esterification:

$$HOCH_2.COOH + ROH \rightarrow HOCH_2.COOR + H_2O$$

and then reacted with hydrogen at high pressure and high temperature in the presence of chromite catalyst:

$$HOCH_2.COOR + 2H_2 \rightarrow HOCH_2.CH_2OH + ROH$$

The yield is about 75%

The duPont & Co in 1940 put in operation a unique plant at Belle, West Virginia, in which glycol was manufd by hydrogenation of methylglycolate, the raw materials for which included its own high pressure synthesis products: formaldehyde and methanol

One US plant manufd, since about 1948, Gc starting with petroleum, chlorine and caustic soda. At first a mixt of ethylene- and propylene oxides was obtd and this was treated with Na hypochlorite (obtd from $NaOH+Cl_2$) and then hydrolyzed with NaOH (Ref 15)

Properties of Ethyleneglycol (Refs 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 14, 15, 16 & 17)

Acid Dissociation Constant at 19°C – $Ka = 5.7 \times 10^{-15}$

Azeotropes of Gc are listed in Ref 15, p 30

Coefficient of Expansion at 20° – 0.00062/°C (Ref 15, p 28)

Dielectric Constant at 20° and 150 meters – 38.66esu (Ref 15, p 28)

Electrical Conductivity at 25° – 1.07×10^{6}mhos (Ref 15, p 28)

Entropy of Formation at 25° – 39.9kcal/mol/°C (Ref 15, p 28)

Flammability Values – See Ref 15, p 39

Germicidal Value – As a preservative against bacterial yeast, or mold growth, it showed a close approach to ethanol and was superior to glycerol (Ref 2)

Heat of Combustion at C_p & 20° – 283.3kcal/mol (Ref 15, p 28)

Heat of Combustion at C_v & 20° – 285.0kcal/mol (Ref 15, p 42)

Heat of Dilution – on mixing Gc with water some contraction with evolution of heat takes place; for example, on mixing one mol of Gc with 2 mols of water 0.60 cals are evolved with formation of $C_2H_4(OH)_2.2H_2O$. It was also reported that on mixing 37% of Gc with 63% of water a hexahydrate was formed (Ref 2)

Heat of Formation at 20° – minus 108.1kcal/mol (Ref 15, p 28)

Heat of Fusion – 44.76cal/g or 2.66kcal/mol (Ref 15, p 28)

Heat of Vaporization at 760mm – 191cal/g

Hygroscopicity – very high; increase in wt 30% in one week or 60% in 2 weeks with formation of dihydrate (See also Ref 15, p 44)

Infrared Absorption Spectra. See Ref 15, p 27

Molal Entropy at 25° – minus 105.4kcal/mol/°C (Ref 15, p 28)

Solubilities of Gc in Various Solvents per 100g, at 25°C: completely miscible with w, lower aliphatic alcs, aldehydes & ketones; AcOH, pyridine, chlorobenz, phenol, furfuraldehyde and glycerol; slightly dissolves in CS_2, chlf & xylene; solubilities in other solvents: DBuPh 0.5, toluene 3.1, o-DCPBz 4.7, CCl_4 6.6 and eth 8.9%. Insol in Gc are NC, cellulose acetate, paraffin oil & other oils, rubber & urea; sol in animal glue and dextrin contg 10% water. Solubilities in 100g Gc at 25° of some inorg salts: K_2SO_4–insol, $KClO_3$ 1.21, $KClO_4$ 1.03, KCl 5.18, NaCl 7.1, $NaClO_3$ 16.0 and $NaClO_4$ 75.5 (See also Ref 15, p 47)

Specific Heat at 20° – 0.561cal/g/°C (Ref 15, pp 29 & 49)

Spontaneous Ignition Temperature – 775°F (413°C) (Ref 15, p 39)

Surface Tension at 20° – 48.4 dynes/cm (Ref 15, p 29)

Thermal Conductivity at 20° – 0.000690 cal-cm/sec/cm^2/°C

Thermal Stability. See Initial Decomposition Temperature

Toxicity. Hunt (Ref 6) claimed that toxicity of Gc is high and might be fatal. This is due to oxidation of Gc to very poisonous oxalic acid which takes place in the body, not immediately, but after a while. It is recommended by Hunt to forbid using Gc in foods. Lethal dose is considered to be 100ml. Hunslick (Ref 5) considers that lethal doses are higher than 100ml. More detailed discussion on toxicity is given in Ref 15, pp 300–302 and in Ref 15a

Uses of Gc. Besides use as a source of manuf of the expl Ethyleneglycol Dinitrate and of some other derivatives, it has been used on a large scale as an antifreeze liquid for automobile cooling systems. One of such materials is known as "Prestone". Gc has also been used in automobile brake fluids and in solvent mixts for various products

Vapor Pressure at 20° – 0.06mm Hg (Ref 15, p 29) at 60° 1.6, and 100° 25mm (Ref 15a)

Viscosity (absol) at 20° – 20.93cp (Ref 15, p 29)

Viscosity in centipoises. See Ref 15, p 57

Under the name of *Ethyleneglycol Condensation Polymers*, Curme & Johnston (Ref 15) placed glycols with several ethylene groups such as diethyleneglycol (described in Vol 5, p D1231-R), triethyleneglycol, $HOCH_2.CH_2.OCH_2.CH_2.OCH_2.CH_2OH$, and numerous group of compds known as Polyethyleneglycols, which are described below

Only lower members of polyethyleneglycols are of interest from the point of view of prepn of expls

Curme & Johnston (Ref 15) describe these compds on pp 153–202 and give their toxicities on pp 302–317

Under the name of *Ethyleneglycol Derivatives*, A.B. Boese et al discussed in the book of Curme & Johnston (Ref 15, pp 114–52 with 415 refs) esters, ethers, ether-esters, alkyds, acetals, ketals, etc of Gc. Their toxicities are given by H.F. Smyth, Jr in the same book on pp 305–17

E. Vernazza discussed in Industria Chimica, Vol 4, pp 990–999 (1929) some expl esters of ethyleneglycol

Nitration of ethyleneglycol, which can be carried out in equipment used for the nitration of glycerin, was discussed by B.P.

Enoksson in SwedP 154069(1956) & CA **50**, 14230(1956) and its kinetics was discussed by J. Roth, F.S. Stow, Jr & D.L. Kouba in IEC **50**, Sept 1958, p 1283 & CA **53**, 5125(1959) *Refs for Glycol:* 1) Beil **1**, 465, (242), [514] & {2052} 2) C.A. Taylor & Wm.H. Rinkenbach, IEC **18**, 676–82(1926) (An evaluation of available information on the physical properties of ethyleneglycol) (27 refs) 3) Ph Naoún, "Nitroglycerine and Nitroglycerine Explosives", translated from Ger by E.M. Symmes, The Williams & Wilkins Co., Baltimore (1928), pp 212-18 4) J.W. Lowrie "Glycerol and the Glycols" Reinhold, NY (1928) (ACS Monograph Series No.44) 5)P.H. Hunslick, IEC **24**, 836 (1932) (Toxicity of Gc) 6) R. Hunt, IEC **24**, 836-37 (1932) 7)A. Stettbacher, "Schiess und Sprengstoffe", Barth, Leipzig (1933), 169 (Glykol) 7a) H. Moureau et al, MP **28**, 252-64 (Prepn of glycols including EtGc) 7b) V. Grignard, Edit, "Traité de Chimie Organique", Masson, Paris, Vol **6** (1940), 1-380 8) C.S. Cragoe, "Properties of Ethyleneglycol and its Aqueous Solutions", USBurStandards, Washington, DC (1943) 9) T.L. Davis, "The Chemistry of Powder and Explosives", J.Wiley ,NY (1943),224 10) A. Pérez Ara, "Tratado de Explosivos", Editorial Cultural, La Habana (Cuba) (1945) (Glicol) 11) Carbon and Carbon Chemical Corp, "Synthetic Organic Chemicals" New York (1946) 12) M. Vivas, R. Feigenspan & F. Ladreda, "Pólvoras y Explosivos Modernos", J. Morata, Madrid, Vol **2**, (1946), 37 (Dietilenoglicol) 12a) E.Whitworth & J.Hornell, Brit P595443 (1947) 13) R.F. Messing, Chem Ind **67**, 41 (1950) (Ethyleneglycol) 13a) L.T. Fairhall, Ind Hyg Newsletter, USPubHealth Service, **7**, No 10,13-14 (1947) (Toxicity of Gc) 14) W.L. Faith D.B. Keyes & R.L.Clark, "Industrial Chemicals", J.Wiley, NY (1950), 327-33 15) G.O.Curme,Jr. & F. Johnston, Editors, "Glycols", Reinhold, NY (1952) (ACS Monograph Series No 114), pp 1-73, 300-302 & 328–350) 15a) T.E. Jordan, "Vapor Pressures of Organic Compounds", Interscience, NY (1954) Chap 3, p 72, Plate 10 16) M. Giua, "Trattato di Chimica Industriale", **VI**, (1), UTET, Torino (1959), 124 (Glicol etilenico) 17) A. & E.Rose (formerly F.M.Turner), "The Condensed Chemical Dictionary", Reinhold, NY (1961), 466-R 18) K&O **10**, (1966), pp 638-646 19) Sax, 3rd edit (1968), 745 (Ethyleneglycol)

Ethyleneglycol: Analysis and Test Methods.

Hillenbrand describes in Ref 1, pp 328-51 the following subjects: 1) Qualitative Identification, 2) Quantitative Determination, and 3) Specifications and Methods of Testing Commercial Glycols

1. Qualitative Identification includes *A. Colorimetric Tests:* a) Oxidation with bromine or permanganate (p328); b)Oxidation with nitric acid (p329); c)Oxidation with periodic acid (p329); d)Reaction with certain phenols (p330)

B. Preparation of Solid Derivatives, such as: a)Esters of benzoic acid and nitrobenzoic acids (p331); b)Alkyl N-Arylcarbamates (Phenyl- and α-Naphthylurethanes) (p331); and c)Bis (triphenylmethyl) Esters (p332)

2. Quantitative Determinations include: *A. Acylation Methods by:* a) Acetic anhydride-pyridine reagent (p333) b) Acetyl chloride (p335) and c) Phthalic anhydride (p336)

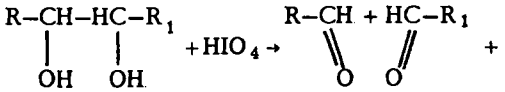
B. Oxidation Methods by: a) Periodic acid (p337); b) Lead tetraacetate (p339); c) Potassium dichromate (p340); and d) Potassium permanganate (p342)

C. Miscellaneous Methods: a)Reaction with hydriodic acid (p342); b) Reaction with ammonium hexanitrocerate reagent (p343); c) Precipitation with silico-tungstic or phosphomolybdic acid (p344); and d) Determination as the ditrityl ether (p345)

3. Specifications and Methods of Testing Commercial Glycols include (pp345-50):

a) Specific gravity 1.1151 - 1.1156; b) Distillation at 760 mm: initial pp min 193°, dry point max 210°; c) Acidity % by wt as AcOH, max 0.01%; d) Moisture, % by wt, max 0.30%; e) Water solubility at 25° – complete; f) Ash, g/100ml, max 0.005%; g) Color, Pt-Co(APHA) units, max 15; h) Odor – mild; i) Suspended matter – substantially free

In Ref 2 is given the following general equation of oxidation of glycols (compounds with adjacent hydroxyl groups) by periodic acid:



$H_2O + HNO_3$

In Ref 3, under "Ethanol Determination by Estimation of Its Hydroxyl Groups Using Various Esterification Procedures" are described methods which are applicable to analysis of glycols
(See also this Vol of Encycl under "Ethanol Determination by Estimation of Its Hydroxyl Groups Using Various Esterification Procedures")
Refs: 1) G.O. Curme, Jr & F. Johnston, Editors, "Glycols", Reinhold, NY (1952), pp 328–51: E.F. Hillenbrand, Jr, "Analysis and Test Methods" 2) S. Siggia, "Quantitative Organic Analysis Via Functional Groups", J. Wiley, NY (1954), pp 16–18; W.D. Pohle et al, "Determination of Compounds with Adjacent Hydroxyl Groups (Glycols)" 3) F.J. Welcher, Editor, "Standard Methods of Chemical Analysis", Vol 2,, Part A (1963), VanNostrand, Princeton, NJ, pp 455–57: "General Methods Using Esterification with Acetic Anhydride"; 458: "Esterification Using Phthalic Anhydride"; 458–59: "Esterification Using Pyromellitic Dianhydride"; 459–61: "Determination of Compounds with Adjacent Hydroxyl Groups (Glycols)" (See Ref 2)

Ethyleneglycol (Technical) for use as a heat-transfer fluid and laboratory testing reagent, is covered by US Military Specification MIL-E-9500A, 21 June 1968 and Amendment 1, 24 June 1969

Chemical Requirements:

1) Chlorides – No ppt present when examined as in Test 4.3.1

2) Total glycol, minimum 99.5% when examined as in Test 4.3.2

3) Acidity (calcd as AcOH), maximum when examined as in Test 4.3.3

4) Ash residue: max 0.005g in 100ml when examined as in Test 4.3.4

5) Distillation: when examined as in Test 4.3.5
Initial bp 192°C, min
95% distilled, 200°, max
Dry point, 208°, max

6) Water solubility at 25°(77°F) when examined as in Test 4.3.6 – miscible with water in all proportions

Physical requirements:

7) Color, not more than 15 on Pt-Co color scale when examined as in Test 4.3.7

8) Flash point – no lower than 116°(240°F) when examined as in Test 4.3.8

9) Odor, mild to the degree to be not objectionable when examined as in Test 4.3.9

10) Specific gravity 1.1151 to 1.1156 at 20/20°C when examined as in Test 4.3.10

11) Suspended matter. Appearance shall be clear; material must be free of suspended matter when examined visually by transmitted light as described in Test 4.3.11

Tests. Water, in accordance with ASTM D1193-66, and reagent grade chemicals shall be used for testing. Blanks shall be run and corrections applied when significant

Tests shall be conducted as follows:

4.3.1 Chlorides. Place 10ml of the material in a 30ml test tube. Add 10ml of water and mix. Add 10 drops of concd nitric acid, agitate the mixture, and filter. Add 10 drops of a 3 percent aqueous soln of silver nitrate to the filtrate. No precipitate shall settle out within 30 minutes after the addition of silver nitrate soln. A slight turbidity will not be cause for rejection

4.3.2 Water content. Water content shall be detd in accordance with Federal Test Method Standard No 791 Test Method No 3253 (ASTM D1744-64) except 10 to 40 grams (9 to 35ml) of sample, depending on the anticipated moisture content, shall be used

4.3.3 Acidity. Acidity shall be determined by measuring 108ml (120g) of sample in a graduate and transferring it to a 250-ml Erlenmeyer flask. A few drops of 1.0% alcoholic solution of phenolphthalein indicator shall be added and the sample titrated with standard 0.1N alc KOH to the first pink end point permanent for 15 secs. The calculation is as follows:

ml KOH x 0.005= Acidity, percent by weight as acetic acid

4.3.4 Ash. Ash residue shall be determined by measuring 50 ml of the sample in a graduate and transferring to a 125 ml platinum dish which has been ignited to constant weight, cooled in a desiccator, and tared to the nearest 0.1 mg. The dish shall be heated until the vapors continue to

burn after the flame is withdrawn. The combustion shall be protected from drafts and the vapors allowed to burn spontaneously until the liquid is consumed. The dish shall then be ignited to a dull red heat, allowed to cool in a desiccator, and weighed to the nearest 0.1 mg. The ash residue per 100 ml shall be calculated as follows:

g ash per 100 ml = g residue x 2

4.3.5 Distillation. Distillation shall be conducted according to Federal Test Method Standard No 141 Test Method No 4301.1, except that distillation shall be conducted at a pressure of 760 mm of mercury or corrected thereto by adding 0.043°C for each mm under 760 mm or subtracting for every mm over 760 mm

4.3.6 Miscibility with water (Water Solubility). Miscibility with water shall be determined by transferring 25 ml of the sample at 25°C(77°F) to a 100 ml glass stoppered graduate and adding 25 ml of water at 25°C in 5 ml portions, shaking the graduate well after each addition. Twenty-five ml of the sample shall be added to 25 ml of water in the same manner. If there is no cloudiness, stratification, or turbidity at any time, the sample shall be considered completely miscible

4.3.7 Color. Color shall be determined in accordance with ASTM D2108-64

4.3.8 Flash point. The flash point of the material shall be determined according to Federal Test Method Standard No 791 Test Method No 1103.6(ASTM D92-66), except that the fire point need not be determined

4.3.9 Odor. Odor shall be determined by pouring 5 ml of the sample on clean filter paper and observing the odor at once

4.3.10 Specific gravity. Specific gravity of the material shall be detd for 20/20°C according to Federal Test Method Standard No 791 Test Method No 402.2 (ASTM D941-55)

4.3.11 Suspended matter. Place 500ml of the sample in a 1000-ml stoppered bottle, and let set for 2 hours. Invert the container, hold up to a well-lighted background, and observe for any suspended matter. There shall be no suspended matter visible in the sample

Ethyleneglycol Polymers; Polyethyleneglycols or Polyglycols.

Curme and Johnston (Ref 13, pp 176–202) assign this name to any ethyleneglycol condensation polymer higher than triethyleneglycol. This includes several *liquid* polymers, such as tetraethyleneglycol and polyethyleneglycols 200, 300, 400 and 600, which are manufactured in this country (The numbers 200 etc signify the average molecular weights of these compds). The *solid* polymers are known as "Carbowax" 1000, 1500, 1540, 4000, 6000, if manufd by the Carbide and Carbon Co, and as "Polyglycol" E1200, E4000, if produced by the Dow Chemical Co

In Germany, polyethyleneglycols are known as *Polyglykol P-*, with a numerical suffix that refers to the units of ethylene oxide involved in its manuf. For instance, Polyglykol P-6, P-8, P-9 and P-12 are yellow, viscous liquids; P-18 a semi-solid brown wax and P-60 & P-80 yellow waxes. The so-called "Oxydwachs" has an approximate molecular wt of 4000 and is offered commercially

The first known polyethyleneglycol was prepd by Lourenco (Ref 2a) by heating a mixture of ethyleneglycol and ethylenedibromide in a sealed tube at 115° to 120°. This glycol corresponded approx to hexaethyleneglycol. In the same year, Wurtz (pronounced Würtz) (Ref 26) obtd some polyethyleneglycols by heating ethyleneglycol, ethylene oxide and water for several weeks. More work on polyethylene was done later by Wurtz (Refs 3 & 4). Many methods of prepn of polyclycols are given in Curme and Johnston's book (Ref 13), among which are the method of Staudinger & Schweitzer (Ref 5) involving the polymerization of ethylene oxide under the influence of catalysts, such as stannic chloride. Staudinger & Lohmann (Ref 6) detd rates of polymerization with various catalysts (See Table 7.9 in Ref 13, p 178). Matignon et al (Ref 6a) prepd polyethyleneglycols by hydration of ethylene oxide in w contg 0.5% sulfuric acid as catalyst. Perry & Hibbert (Ref 8) described the first direct method for synthesizing a single polyethyleneglycol. The method involved the reaction between the dichloride of a pure lower-molecular-weight polyglycol and two mols of the monosodium alcoholate of another pure polyglycol. Using this procedure, polyglycols with as high as 186 ethylene groups were obtained (See also Refs 9 & 10)

Commercial methods of prepn of polyglycols are described by Ellis (Ref 7) and by Curme and Johnston (Ref 13). One of the American methods consisted of adding to water and ethyleneglycol (or to diethyleneglycol containing a catalytic proportion of NaOH), ethylene oxide at such a rate as to maintain the temperature of the liquid between 120° and 135°. The system was then maintained at the above temperature for 2 to 5 hours at a pressure of 60 psi; the reactor was cooled with water as some heat was liberated during the reaction. The mixture was then neutralized and filtered

Note: The reaction was considered completed when the pressure in the system at constant temperature remained unchanged for at least one hour

The German method of prepn was described by Brandner and Goepp (Ref 11)

The physical properties of polyethyleneglycols vary with molecular weight, either increasing or decreasing. Such properties as freezing point, flash point, density and viscosity increase with an increase in molecular weight, while the vapor pressure, hygroscopicity and solubility in organic compounds decrease

All the polyglycols are soluble in water and are miscible with many waxes, gums, oils, starches and organic solvents. Properties of various polyglycols are given on pp 181 to 185 of Ref 13

Uses: Polyglycols are widely used in cosmetics, resins, plasticizers, textiles, agriculture, coatings, in electronics and as lubricants for rubber molds, textile fibers, etc

Toxicity

Liquid polyethyleneglycols 200, 300 and 400 are about as toxic as triethyleneglycol, but those with higher molecular weights are less toxic

Solid polyethyleneglycols are less toxic than liquid ones and their toxicity decreases with increase in molecular weight

Analysis and Tests

Curme and Johnston's book, pp 328–350 gives specifications and methods of analysis of polyethyleneglycols as high as triethyleneglycol but not for higher ones

Refs: 1) Beil – not listed in the alphabetical index 2a) A.V. Lourenco (1822–1893), CR **49**, 619(1859) 2b) A. Wurtz, CR **49**, 813(1859) 3) A. Wurtz, AnnChimPhys (3) **69**, 330(1863) 4) A. Wurtz, Ber **10**, 90(1877); Bull(Fr)(2), **29**, 530(1878) 5) H. Staudinger & O. Schweitzer, Ber **62**, 2395(1923) 6) H. Staudinger & H. Lohmann, Ann **505**, 41(1933) 6a) C. Matignon et al, Bull(Fr), (5) **1**(1934), p 1308 7) C. Ellis, "The Chemistry of Synthetic Resins" Reinhold, NY, V2(1935) 8) S.Z. Perry & H. Hibbert, CanJResearch **B14**, 77(1936) 9) R. Fordyce, E.L. Lovell & H. Hibbert, JACS **61**, 1905(1939) 10) R. Fordyce & H. Hibbert, JACS **61**, 1910(1939) 11) J. D. Bradner & R.M. Goepp, Jr, PB Rept No **85**, 159(1945) 12) Kessler Chemical Co, "Polyethyleneglycol Esters", Philadelphia (1948) 13) G.O. Curme Jr & F. Johnston, Eds, "Glycols", Reinhold, NY(1952), pp 176–193 (Polyethylene Glycols) 14) Sax, 3rd edit(1968), 1035(Polyethylene Glycols)

Ethyleneglycol-Sugar Mixture, Nitrated. In order to prepare a cheaper expl, the Germans used to add about 25% sugar to ethyleneglycol and then nitrate the mixture in the same manner as straight glycol or straight glycerin. The resulting product was a mixture of EGDN and Heptanitrosacchorose. It was a powerful expl with N content 17.8 to 17.9%

Ref: Naoúm, NG(1928), 255

Ethyleneglycol Acetate or **Glycol Monoacetate,** $HO.CH_2.CH_2.OOC.CH_3$; mw 104.10; colorless, nearly odorless liq, sp gr 1.108, bp 181–82°; partially sol in w; sol in alc & eth. It can be prepd from ethyleneglycol and AcOH or by passing ethyleneoxide into hot AcOH contg AcONa or sulfuric acid. Its toxicity is discussed in Ref 2, pp 311–12 and in Ref 4. It is used as a solvent for NC, cellulose acetate or camphor (See also Ethyleneglycol Diacetate)

Refs: 1) Beil **2**, 141, (66) & [154] 2) Curme & Johnston(1952), 101, 129 & 311–12 3) CondChemDict(1961), 467-R 4) Sax, 3rd edit(1968), 747

Ethyleneglycol Acetate Nitrate or **Glycol Monoacetate Mononitrate,** $O_2NO.CH_2.CH_2.OOC.CH_3$; mw 149.10, sp gr 1.29 at 18°, oil, insol in w, sol in dil mineral acids. It was prepd by dissolving glycol monoacetate in nitrosulfuric acid. (See also Ethyleneglycol Diacetate)

Refs: 1) Beil **2**, 142 2) L. Henry, Ann (4) **27**, 259(1872) 3) Curme & Johnston(1952) – not found

Ethyleneglycol-bis(trinitrobutyrate). See Ethyleneglycol-di(trinitrobutyrate)

Ethyleneglycol Butylether, Glycolmonobutyl Ether or **2-Butoxyethanol,** $HO.CH_2.CH_2.OC_4H_9$; mw 118.18, colorless liq with mild odor, sp gr 0.9019 at 20/20°, n_D 1.4190 at 25°, bp 171.2°, fl p 165°F, sol in w & in mineral oils. It can be prepd by many methods described in Ref 3, pp 114–15; industrially it can be prepd by reacting ethylene oxide with butanol, in presence of a catalyst as described in Ref 2. It is used as solvent for NC, resins, spray & brushing lacquers, varnishes etc. Its toxicity is discussed in Ref 3, pp 306 & 309 and in Ref 5

Refs: 1) Beil **1**, [519] & {2081} 2) C.O. Young Jr, USP 1696874(1928) 3) Curme & Johnston(1952), 114–17, 306 & 309 4) CondChemDict(1961), 467-R 5) Sax, 3rd edit (1968), 502 & 745–46

Ethyleneglycol Butylether Acetate or **Glycol Monobutylether Acetate,** $CH_3COO.CH_2.CH_2.OC_4H_9$; mw 160.21; colorless liq with fruity odor, sp gr 0.9424 at 20/20°, n_D 1.420 at 20°, fr p –63.5°, bp 192.2°, fl p(open cup) 190°F; very sl sol in w; sol in hydrocarbons and in org solvents. Prepn not found in CA. Used as a high-boiling solvent for NC, lacquers, epoxy resins, etc

Refs: 1) Beil, not found 2) Curme & Johnston (1952), 138 3) CondChemDict (1961), 468-L 4) Sax, 3rd edit (1968), 502 (Butyl "Cellosolve" Acetate)

Ethyleneglycol Condensation Products, Nitrated. Accdg to Foulon (Ref 2), the Westfälisch-Anhaltische Sprengstoff AG (Ref 1) proposed using the nitrated glycol (such as ethylene- or diethylene-glycol) condensation products as explosive gelatinizers for NC intended for use in proplnts. It was claimed that these nitrated condensation products can gelatinize any kind of NC cold, and for this reason it is not necessary to conduct the extrusion, or rolling operations hot. The resulting NC proplnts are uniform in structure, stable and burn uniformly. No additional solvents are required for gelatinization

Refs: 1) Westfälisch-Anhaltische Sprengstoff AG, Ger P 548427(1931) & CA **26**, 3670(1932) 2) A. Foulon, SS **27**, 400(1932) & MAF **14**, 464(1935)

Ethyleneglycol Diacetate or **Glycol Diacetate,** $CH_3.COO.CH_2.CH_2.OOC.CH_3$; mw 146.14; colorless liq with faint odor, sp gr 1.063 at 20/20°, fr p –41.5°, bp 190.5°, vapor pressure 0.3mm Hg at 20°, fl p 220°F, n_D 1.415 at 20°; soly in w 14.3% at 22°; miscible with alc, ether & benz. It was first prepd in 1859 by Wurtz (pronounced Würtz) from ethylenedibromide (or diiodide) and Ag acetate; Demole prepd it in 1875 from ethylenedibromide and anhydrous K acetate and Seelig patented, in 1887 in Germany, its prepn by boiling for 35 hours a mixture of 74g of ethylenedibromide, 80g of Na acetate and 200g of AcOH. Its industrial prepn on a large scale from derivatives of the chlorination of natural gas was patented in Germany in 1921 by Meyer and in the USA in 1922 by Rodebush (Refs 1 & 2)

In CondChemDict (Ref 4) it is stated that it can be prepd from ethyleneglycol and acetic acid and ethylenedichloride and Na acetate

Method of purification of commercial diacetate, such as supplied by the Carbide and Carbon Corp is described by Taylor & Rinkenbach (Ref 2, p 1305). From the product boiling at 188° to 189.6° at 739.2mm Hg, the water that may have been present was removed by treatment with Ca chloride. The clear filtrate was kept from contact with moist air and fractionally distilled at 125° and 85mm Hg. The middle and largest of 3 fractions, representing ca 70% of the volume treated, was retained for examination of properties of diacetate. It was found that it is neutral, stable, has a high bp, very low volatility at RT, and a very high solvent power for organic expl compds. For these reasons it is suitable for purification of expls (such as TNT) by crystallization. It also can be used for control of their crystal size. It is less toxic than DEGDN (Ref 3, p 311). See also Ref 5. See also Ethyleneglycol Monoacetate

Refs: 1) Beil **2**, 141–42, (66) & [155] 2) C.A. Taylor & Wm H. Rinkenbach, JACS **48**, 1305–09(1926) 3) Curme & Johnston (1952), 101,121,129 & 311 4) CondChemDict (1961), 467-L 5) Sax, 3rd edit (1968), 746

Ethyleneglycol Diacrylate, $(CH_2{=}CHCO_2CH_2)_2$; mw 170.17; bp 53–7°(0.6mm) and 70°(1mm), sp gr 1.0935 at 26/20°, n_D 1.4529(1.4546) at 20°. It is prepd by refluxing ethyleneglycol, acrylic acid, p-toluenesulfonic acid, pyrogallol, and cuprous chloride in benz in the dark; obtd 40% yield. It is used to prepare **Ethyleneglycol di-trinitrobutyrate** (qv)
Refs: 1) Beil, not found 2) J.G. Burtle et al, JOC **17**, 1302–4(1952) & CA **47**, 9913(1953) 3) Kirk & Othmer, Vol 1, (1963), p 289

Ethyleneglycol Diethylether, Glycol Diethylether (GDEE), 1,2- Diethoxyethane (called in Lange **Diethyl Cellosolve**), $C_2H_5O.CH_2.CH_2.OC_2H_5$; mw 118.17, colorless liq with sl ethereal odor, sp gr 0.8417 at 20/20°, fr p –74°, bp 121.4°, fl p 95°F, vapor pressure 9.4mm at 20°; partially miscible with w. It can be prepd by reacting the Na alcoholate of an ethyleneglycol ether with ethyl halide or by reacting diethyl sulfate with the ethyleneglycol monoethyl ether (Refs 2 & 3)

It is used in org synthesis and as solvent; when added to colloidal systems such as detergents and wetting agents of limited water soly, permits w dilution while reducing, without gelling or clouding. Its toxicity is discussed in Ref 3, p 306
Refs: 1) Beil 1, 468, [519] & {2078} 2) A. Wurtz, AnnChimPhys [3] **55**, 431(1859) 3) Curme & Johnston(1952), 119 & 306 4) CondChemDict (1961), 467-L (Ethyleneglycol Diethylether) 5) Sax, 3rd edit (1968), not found

Ethyleneglycol Dilactate or *Glycol Dilactate*, $CH_2.O.CO.CH(OH).CH_3$ | $CH_2.O.CO.CH(OH).CH_3$; mw 216.19. It may be considered as the parent compd of its dinitrate deriv, although not used for its prepn
Ethyleneglycol Dilactate Dinitrate, $CH_2.O.CO.CH(ONO_2)CH_3$ | $CH_2.O.CO.CH(ONO_2)CH_3$; mw 296.19, N 9.46%, OB to CO_2 –64.9%. This compd was prepd during WWII by the duPont Co and found to be an expl stable to heat and extremely insensitive to impact. No description of its prepn was found

In the International Test at 75°, this compd showed no decompn in 48 hrs. Its impact test with 20kg wt was negative at 44.5 inches (Ref 2)
Refs: 1) Beil, not found 2) Blatt, OSRD **2014** (1944)(No prepn given) 3) CA, not found thru 1966

Ethyleneglycol Dimethylether (EGDME) or **Glycol Dimethylether (GDME),** also known as **1,2-Dimethoxyethane** or **Dimethylcellosolve,** $CH_3O.CH_2.CH_2.OCH_3$; mw 90.12; colorless, flammable liq with a mild ether odor; sp gr 0.8683 at 20°, n_D 1.3792 at 20°, fr p –69°, bp 85.2°, fl p (open cup) 34°F; pH 8.2; sol in w and hydrocarbons; vapor pressure 61.2mm at 20°. It can be prepd by reacting Na alcoholate of an ethyleneglycol methylether with methyl halide. Used as a solvent. Its toxicity is discussed in Ref 3, p 306
Refs: 1) Beil 1, 467, (244) & [518] 2) W. Lippert, Ann **276**, 171(1893) 3) Curme & Johnston (1952), 118–19 & 306 4) CondChemDict (1961), 467 5) Sax, 3rd edit (1968), 746

Ethyleneglycol Dinitrate. See under ETHYLENEGLYCOL NITRITES AND NITRATES

Ethyleneglycol Dinitrite. See under ETHYLENEGLYCOL NITRITES AND NITRATES

Ethyleneglycol Diphenylether or **Di(phenyloxy)-ethylene.** See Bis(phenyl)-ethyleneglycol Ether in Vol 2 of Encycl, p B153-R. Its Bis-(dinitrophenyl)-ethyleneglycol ether, also known as [Ethyleneglycol-2,4,2′,4′-tetranitrodiphenyl]-ether is on p B153-R and its Bis(trinitrophenyl)-ethyleneglycol ether, also known as [Ethyleneglycol-2,4,6,2′,4′,6′-hexanitrodiphenyl]-ether is on p B154-L

Addnl refs for these compds are listed under Di(phenyloxy)-ethylene in Vol 5, p D1470-R

Ethyleneglycol-di-trinitrobutyrate (EGTNB) or **Glycol-di-trinitrobutyrate (GTNB),** $CH_2.COO.CH_2.CH_2.C(NO_2)_3$ | $CH_2.COO.CH_2.CH_2.C(NO_2)_3$; mw 472.24,

N 17.80%, OB to CO_2 −33.9%, OB to CO ±0%; crysts, sp gr 1.63, mp 95–96°. It can be prepd by the addition of nitroform to ethyleneglycol diacrylate. Expln temp 230° in 5 seconds, detonation rate 7340m/sec at d 1.63 and sensitivity to impact about that of Comp B. The low mp of EGTNB suggests its use as casting medium for HE compds

Refs: 1) Beil, not found 2) Anon, US Rubber Co, Quarterly Progress Rept No **14**, Feb 1 to May 1, 1951, p 9 (Contract NOrd 10129) 3) W.F. Sager & D.V. Sickman, NAVORD Rept **483**(1952) (Research and Development in New Chemical High Explosives)

Ethyleneglycol Ethylether, Glycol Monoethylether, Cellosolve or **2-Ethoxy-ethanol-1,** $HO.CH_2.CH_2.OC_2H_5$; mw 90.12; colorless, nearly odorless liquid, sp gr 0.9311 at 20/20°, n_D 1.4060 at 25°, fr p −70°, bp 135.1°, fl p 130°F; miscible with w and hydrocarbons. It can be prepd by reacting ethylene oxide with ethanol in presence of a catalyst or by other methods. Its toxicity is discussed in Ref 4, pp 306 & 308 and in Ref 6. It is used as solvent for NC, resins, oils, etc (See Ref 5). It was proposed (Ref 3) in mixture with perchloric acid, as a liquid serving for destruction of land-mines

Refs: 1) Beil **1**, 467, (244) & [518] 2) M.H. Palomaa, Ber **42**, 3876(1909) 3) J.H. Frazer et al, USP 2504119(1950) & CA **44**, 6130(1950) 4) Curme & Johnston (1952), 114–17, 306 & 308 5) CondChemDict (1961), 468-L 6) Sax, 3rd edit (1968), 747

Ethyleneglycol Ethylether Acetate, Glycol Monoethylether Acetate or **"Cellosolve" Acetate,** $CH_3COO.CH_2.CH_2.OC_2H_5$; mw 132.16; colorless liq with mild ether-like odor; sp gr 0.9748 at 20/20°, n_D 1.4030 at 25°; fr p −61.7°, bp 156.3°, fl p 135°F; sol in w (20%) and miscible with alc, eth, acet and aromatic hydrocarbons; can be prepd by treating ethyleneglycol monoethylether with acetyl chloride. Its toxicity is discussed in Ref 3, p 313. It is used as solvent for NC, oils & resins; removes varnish and retards "blushing" in lacquers

Refs: 1) Beil **2**, 141, (66) & [155] 2) L. Henry, ChemZentr **1902** II, 1403 3) Curme & Johnston (1952), 138 & 313 4) CondChemDict (1961), 468-L

Ethyleneglycolformal or **Ethyleneglycol Methylene Ether.** See 1,3-Dioxalane in Vol 5, pp D1405-R to D1406-L

Ethyleneglycolglycolate Dinitrate, $(O_2NO)CH_2.CH_2.O.C(:O).CH_2(ONO_2)$; mw 210.10, N 13.33%, OB to CO_2 −22.8%. It is a liquid expl prepd and examined during WWII by the duPont & Co and found to be less sensitive to impact than NG and of good stability (23 minutes in 65.5°C KI Test)

Refs: 1) Beil, not found 2) Blatt, OSRD **2014**(1944) (No prepn given) 3) Curme & Johnston (1952), not found

Ethyleneglycolglucoside and Derivatives

Ethyleneglycol-mono-glucoside (EGG), $HOCH_2.CH_2O.\underline{CHOCH(CH_2OH)CHOH.CHOH.CHOH}$; mw 224.21, α-form (ndls from alc-EtAc), sol in water & alc, hygroscopic. It is prepd from d-glucose, glycol, water and α-glucosidase from bottom-fermented, air-dried yeast (Ref 3), β-form (cryst from alc-eth), mp 137–8°, sol in water & alc; prepd from d-glucose, glycol, water and emulsin (Ref 2)

Ethyleneglycol-di-glucoside, $_2\{CH_2O.\underline{CHOCH(CH_2OH)CHOH.CHOH.CHOH}\}$; mw 386.36, β-form, cryst, mp 131.8°. It was prepd from glycol, glucose, water and emulsin (Ref 4)

Refs: 1) Beil **31**, (193–4) 2) E. Bourquelot & M. Bridel, CR **158**, 898(1914) 3) Ibid, CR **158**, 1219(1914) 4) Ibid, CR **168**, 253, 1018(1919)

Ethyleneglycolglucoside Nitrate (EGGN). It is prepd as a mixture with NG & EGDN by standard nitration procedure of a mixture of glycerol glycol & ethyleneglycol glucoside (commercial, contg polyethyleneglycols, mono- or diglucoside unspecified). The oily product is useful for dynamite formulation

Refs: 1) Beil, not found 2) A.A. Young, USP 2602733(1952) & CA **46**, 9312(1952)

Ethyleneglycol Lactate or **Glycol Monolactate,** $CH_2.OC(:O)CH(OH).CH_3$; CH_2OH mw 134.13, bp 285–7°, 124° (4.2mm), 100° (0.4mm), sp gr 1.1996 at 20°, n_D 1.4495 at 20°. It was first prepd by heating the glycol with lactic acid in the presence of a phosphoric catalyst; later prepd using the aluminum isopropoxide exchange with methyl lactate; most recently using the glycol, the acid and a cation exchange resin in the acid form

Refs: 1) Beil, not found 2) A.G. Evdokimov, Priroda **1940**, No 11, 68–70 & CA **38**, 718(1944) 3) M.L. Fein & C.H. Fisher, JACS **68**, 2631–2 (1946) & CA **41**, 1612(1947) 4) H. Spaenig et al, GerP 1912730(1970) & CA **73**, P120114 (1970)

Ethyleneglycol Lactate Dinitrate or **Glycol Monolactate Dinitrate** $CH_2.OC(:O)CH(ONO_2)CH_3$; mw 224.12, N 12.50%, OB to CO_2 –42.8%. $CH_2.ONO_2$ It was prepd by duPont & Co by, presumably, standard nitration of the lactate ester and found, on examination, to be an explosive stable at 75° but decomposing at 105° with evoln, after 17 hours, of 30cc of gas, vs 120cc for NG. Its impact sensitivity with 20kg wt was 40 inches for 1/10 shots and 37 inches for 0/10 shots

Refs: 1) Beil, not found 2) CA, not found 3) Blatt, OSRD **2014**(1944)(Prepn not given)

Ethyleneglycol Methylether, Glycol Monomethyl Ether, 2-Methoxyethanol or **Methyl Cellosolve,** $HO.CH_2.CH_2.OCH_3$; mw 76.09; colorless liq with mild agreeable odor; stable; sp gr 0.9663 at 20/20°, n_D 1.4021 at 20°, fr p –85.1°, bp 124.6°, fl p 115°F; miscible with w, alc, ketones, glycols & hydrocarbons. It can be prepd by heating sodium glycocide with methyl iodide. Its toxicity is discussed in Ref 5. It is used as solvent for NC, cellulose acetate, resins, etc

Refs: 1) Beil **1**, 467, (244) & [518] 2) M.H. Palomaa, Ber **35**, 3300(1902) 3) Curme & Johnston (1952), 116–17, 141 & 306–08 4) CondChemDict (1961), 468-R 5) Sax, 3rd edit (1968), 747–48

Ethyleneglycol Methyleneether. Same as Ethyleneglycolformal

Ethyleneglycol Methylether Acetate or **Glycol Monomethylether Acetate,** $CH_3COO.CH_2.CH_2.OCH_3$; mw 118.13; colorless liq with pleasant ester odor; stable; sp gr 1.0067 at 20/20°, fr p –65.1°, bp 145.1°, fl p 140°F; sol in w, miscible with the common org solvents. It can be prepd by treating ethyleneglycol methylether with AcCl and pyridine. Its toxicity is discussed in Ref 3, p 313 and in Ref 5. It is used as solvent for NC, cellulose acetate, gums, resins, waxes and oils

Refs: 1) Beil **2**, 141, (66) & [154] 2) M.H. Palomaa, Ber **42**, 3875(1909) 3) Curme & Johnston (1952), 138 & 313 4) CondChemDict(1961), 468-R 5) Sax, 3rd edit (1968), 917 (Methyl "Cellosolve" Acetate)

Ethyleneglycol Monoacetate. See Ethyleneglycol Acetate in this Vol

Ethyleneglycol Monoacetate Nitrate. See Ethyleneglycol Acetate Nitrate in this Vol

Ethyleneglycol Monoalkyl Ethers are compds of general formula $RO.CH_2.CH_2.OH$, where R stands for methyl, ethyl, proply, butyl, etc radicals. They are described here as Ethyleneglycol Butylether, Ethyleneglycol Ethylether and Ethyleneglycol Methylether. Their toxicities are described in the following refs:

1) H.W. Werner, J.L. Mitchell, J.W. Miller & W.F. von Oettingen, "The Acute Toxicity of Vapors of Several Monoalkyl Ethers of Ethylene Glycol", Journal of Industrial Hygiene and Toxicology, **25** (4), April 1943, pp 157–63(12 refs)

2) H.W. Werner et al, "Effects of Repeated Exposures of Rats to Vapors of Monoalkyl Ethylene Glycol Ethers", JIndHygToxicol **25**(8), Oct 1943, pp 374–79(8 refs)

3) H.W. Werner et al, "Effects of Repeated Exposure of Dogs to Monoalkyl Ethylene Glycol Ether Vapors", JIndHygToxicol **25** (9), Nov 1943, pp 409–14(12 refs)

4) Curme & Johnston (1952), 305–09 (Toxicity

of alkyl ethers of Ethyleneglycol)
5) Sax, 3rd edit (1968) (Under compd name)

Ethyleneglycol Monobutylether. See Ethyleneglycol Butylether

Ethyleneglycol Monobutylether Acetate. See Ethyleneglycol Butylether Acetate

Ethyleneglycol Monoethylether. See Ethyleneglycol Ethylether

Ethyleneglycol Monoethylether Acetate. See Ethyleneglycol Ethylether Acetate

Ethyleneglycol Monolactate. See Ethyleneblycol Lactate

Ethyleneglycol Monolactate Dinitrate. See Ethyleneglycol Lactate Dinitrate

Ethyleneglycol Monomethylether. See Ethyleneglycol Methylether

Ethyleneglycol Monomethylether Acetate. See Ethyleneglycol Methylether Acetate

Ethyleneglycol Mononitrate. See under ETHYLENEGLYCOL NITRITES AND NITRATES

Ethyleneglycol Mononitrite. See under ETHYLENEGLYCOL NITRITES AND NITRATES

Ethyleneglycol-Mononitrite-Minonitrate. See under ETHYLENEGLYCOL NITRITES AND NITRATES

Ethyleneglycol Monophenylether. See Ethyleneglycol Phenylether

ETHYLENEGLYCOL NITRITES AND NITRATES

Ethyleneglycol Nitrite or *Glycol Mononitrite*, $\begin{matrix}CH_2.OH\\|\\CH_2.ONO\end{matrix}$; mw 91.07, N 15.38%, OB to CO_2 −61.5%. Prepn of this compd is not given in the open literature

Refs: 1) Beil, not found 2) CA, not found

Ethyleneglycol Nitrite-Nitrate or **Glycol Mononitrite-Mononitrate,** $\begin{matrix}CH_2(ONO)\\|\\CH_2(ONO_2)\end{matrix}$; mw 136.06, N 20.59%, OB to CO_2 −11.8%, bp 53–66°(20mm) (Ref 5)

A. Kekulé prepd in 1869 an unstable liquid (known now as Kekulé Oil) when he passed ethylene thru a cooled nitric-sulfuric acid mixture, which he designated as ethyleneglycol nitrate-nitrite. Later, Wieland & Sakellarios (Ref 2) showed that this product was a mixt of ethyleneglycol dinitrate and and the nitric acid ester of 2-Nitroethyl Alcohol (See also Ref 4)

Naoúm (Ref 3) gives the following equation in prepn of nitrite-nitrate from ethylene:

$$\begin{matrix}CH_2\\\|\\CH_2\end{matrix} + 2HNO_3 = \begin{matrix}CH_2.ONO\\|\\CH_2.ONO_2\end{matrix} + H_2O$$

and then the nitration continues with formation of Glycol Dinitrate and nitrous acid:

$$\begin{matrix}CH_2.ONO\\|\\CH_2.ONO_2\end{matrix} + HNO_3 = \begin{matrix}CH_2.ONO_2\\|\\CH_2.ONO_2\end{matrix} + HNO_2$$

Refs: 1) Beil **1**, 469 & [521] 2) H. Wieland & E. Sakellarios, Ber **53B**, 203(1920) 3) Naoúm, NG(1928), 221 4) Curme & Johnston (1952), 130 & CA **50**, 11938(1956) 5) G. Rossmy, Ber **88**, 1969–74(1955) (Prepd from ethylene oxide & NO_2 in chlf at 2–3°)

Ethyleneglycol Nitrate, Glycol Mononitrate, Nitro-1,2-ethandiol or **Mononitroglycol,** $HO.CH_2.CH_2(ONO_2)$; mw 107.07, N 13.08%, OB to CO_2 −37.4%; colorless, somewhat volatile liquid, with faint aromatic odor; sp gr 1.348 at 20/4°, fr p below −20°, bp 91–2° in a vacuum of a water jet pump and decomp at atm pressure; miscible with w and sol in alc, eth & chlf; dissolves in concd sulfuric acid with considerable evolution of heat, so that in the detn of the nitrogen content by Nitrometer Method (See Vol 1 of Encycl, pp A373–A378)

it should be first dissolved in moderately strong sulfuric acid and then concd sulfuric acid added gradually

It was first prepd in 1872 by Henry (Refs 1 & 2) by the action of Ag nitrate on bromohydrin. It can also be prepd by nitration of glycol with straight concd nitric acid (free of N oxides) as described by Naoúm (Ref 3, p 227). In industry it is formed as a by-product inthe manuf of Dinitroglycol. It is slightly more viscous than DNGc and is a good gelatinizer for NC. It is a rather powerful explosive, but has not been considered for commercial expl compns because it has no advantage over the dinitrate and, moreover, its manuf is more difficult and its high soly in water is a serious disadvantage. Its heat of expln is 943.7cal/g at C_v with water liq or 855.6 with water gas; molecular heat of formation 90.0kcal and Trauzl Test value 375cc (in glass tube with sand tamping) against 650cc for Dinitroglycol. Its toxicity is discussed in Ref 4, p 312.

Refs: 1) Beil **1**, 469 2) L. Henry, AnnChimie et de Physique (4) **27**, p 243(1922) 3) Naoúm, NG (1928), 227–28 4) Curme & Johnston (1952), 133 & 312

Addnl Refs for EGMN: A) V. Öhman, Ing-VetenskapsAkadHandl No **139**, 48pp(1936) & CA **30**, 5559(1936) B) W.F. Von Oettingen, NatlInstHealthBull No **186**, 76pp(1946) & CA **41**, 809(1947) (Toxicology)

Ethyleneglycol Dinitrite, $(ONO)CH_2.CH_2(ONO)$; mw 120.07, N 23.32%, OB to CO_2 −26.7%; colorless liq, sp gr 1.2156 at 0°, fr p −15°, bp 96–98°, n_D 1.3884 at 18.5°, toxic; insol in w; sol in eth, benz & acet; reacts with ethanol to form ethyl nitrite. On standing it decomposes to oxalic acid and decompd by alkalies. It can be obtd by distilling Glycerol Trinitrite with somewhat more than theoretical quantity of ethyleneglycol (Ref 3) or by treating the glycol with HNO_2 (Ref 4). Its expl properties were not reported

Refs: 1) Beil **1**, 469 & {2111} 2) G. Bertoni, GazzChimItal **15**, 353(1885) 3) Curme & Johnston (1952), 133 4) B.N. Kondrikov, TeoriyaVzryvchatykhVeshchestv, SbStatei **1963**, 296–337 & CA **60**, 2713(1964)

ETHYLENEGLYCOL DINITRATE (EGDN or EGcDN), Nitroglycol (NGc), Dinitroglycol, Dinitrodimethylene or **Ethylene Dinitrate,**

$$\begin{matrix} CH_2.ONO_2 \\ | \\ CH_2.ONO_2 \end{matrix}$$

; mw 152.07, N 18.42%, OB to CO_2 ±0%, OB to CO +21%; colorless volatile liquid when in pure state; yellowish liquid in crude state; sp gr 1.488 at 20/4° or 1.480 at 25°; n_D 1.4452 at 25° or 1.4472 at 20°, fr p −22.75° (vs +13.1° for NG); fr p given in Ref 19, p 132 is −22.3°; bp 199° at 760mm Hg (with decompn). Wieland & Sakellarios (Ref 3a as quoted in Ref 5, p 1499) found EGDN to boil at 105.5° under a pressure of 19mm Hg, but when quickly heated under atm pressure it exploded at 215°. This was preceded by partial decompn analogous to that found in case of NG

Its solubility and other props are given after description of its methods of prepn

L. Henry was the first to prep EGDN in a pure state (in 1870) by dropping small portions of glycol into a mixture of nitric and sulfuric acids cooled to 0°, but A. Kekulé previously (1869) nitrated ethylene and obtd an unstable oil which he presumed to be Glycol Nitrite-Nitrate but which in 1920 was shown by Wieland & Sakellarios to be a mixture of EGDN and the nitric ester of β-Nitroethyl Alcohol (quoted from Ref 5, p 1195 and from Refs 1, 6 & 7). Other investigators preparing NGc before publication in 1926 of Rinkenbach's work (Ref 5) included: Champion (1871), Neff(1899) & Wieland & Sakellarios(1920) (quoted from Ref 5) and also Dautriche (Ref 2), Hough (Ref 3) & Oehme (Ref 4). Rinkenbach (Ref 5, p 1196) prepd EGDN by nitrating purified glycol obtd by fractionating the commercial product under pressure of 40mm Hg and at temp 120°(See Ref 2, under Ethyleneglycol, p 678). For this 20g of middle fraction of purified glycol was gradually added to a mixt of 70g nitric acid (d 1.42) and 130g of sulfuric acid (d 1.84), maintaining the temp at 23°. The resulting 49g of crude product was washed with 300ml of w (in small portions) to obtain 39.6g of purified product. The low yield so obtd could be improved by maintaining a lower temp and using a different nitrating acid mixture

In the book of Naoúm (Ref 7) are described several laboratory and industrial methods for prepn of EGDN, such as:

1) *Direct Nitration of Glycol* is carried out in

exactly the same manner, with the same apparatus and with the same mixed acids as in the nitration of glycerine, such as described on pp 25–75 of Naoúm. A brief description for prepn of NGc is given in Vol 5, under DYNAMITE, p D1590. In the test nitration of anhydrous glycol (100g) with 625g of mixed acid (HNO_3 40 & H_2SO_4 60%) at 10–12°, the yield was 222g and it dropped to 218g when the temp was raised to 29–30°. When 500g of mixed acid (HNO_3 50 & H_2SO_4 50%) was used at 10–12°, the yield increased to 229g. In commercial nitration, the maximum yields obtd from 100kg anhydrous glycol and 625kg of mixed acid contg HNO_3 41, H_2SO_4 58 & water 1% were 222.2kg NGc at nitrating temp of 10–12° and only 218.3kg at 29–30°. This means 90.6% of theory, as compared to 93.6% with NG. Better results (229kg of NGc or 93.4% of theory) obtd on nitrating with 500kg of acid contg HNO_3 50 & H_2SO_4 50% at 10–12° (Ref 7, p 219)

2) *Direct Production of NGc from Gaseous Ethylene.* This method was introduced after WWI by the Chemische Fabrik Kalk, GmbH in Köln-Kalk by Dr H. Oehme and later patented in the USA (Ref 4). Accdg to description given in Ref 7, pp 220–22, the direct action of mixed acid on gaseous ethylene is similar to the method proposed by Kekulé in 1869 and mentioned above. The action of mixed acid on ethylene can be represented as follows:

$$\underset{\text{Ethylene}}{CH_2=CH_2} \xrightarrow{HNO_3} \underset{\text{Nitroethyl Alcohol}}{CH_2.NO_2.CH_2.OH} \xrightarrow{HNO_3} \underset{\text{Nitroethyl Nitrate}}{CH_2.NO_2.CH_2.ONO_2}$$

and simultaneous reaction:

$$\underset{\text{Ethylene}}{CH_2=CH_2} \xrightarrow{2HNO_3} \underset{\text{Glycol Nitrite-Nitrate}}{CH_2.ONO.CH_2.ONO_2} \xrightarrow{HNO_3} \underset{\text{Glycol Dinitrate}}{CH_2.ONO_2.CH_2.ONO_2} + HNO_2$$

This 2nd reaction, in which a 3rd mole of nitric acid drives out the nitrous acid, explains the rather high content of nitrogen oxides in the spent acid. This mixture of 40–50% Nitroethyl Nitrate & 40–50% Nitroglycol is known as "Kekulé Oil". As its N content is up to 19.5%, which is even higher than for NGc, it is a highly brisant expl. Accdg to GerP 310789 of 1918, such oil can be formed when a current of dry ethylene is passed thru an anhydrous mixed acid contg 32% HNO_3. The yield is 70% of the theoretical calcd on amt of ethylene used. After washing the oil with hot w, it can be converted directly into a Dynamite by taking it up in absorbents like Kieselguhr or gelatinized with NC to form Gelatindynamite. Direct separation of the oil from the spent acid accounts for only two-thirds of the total oil. The spent acids still contain 8 to 10% of the total oil, which can be separated by extraction with chloroform or ethylenechloride. This residual oil is unstable.

Accdg to GerP 386687 of 1923, the neutralization of crude oil after washing with hot w is done with neutral Na sulfite, in order to avoid the formation of emulsions which occur when strong alkalies are used

A simple method of separating NGc from Kekulé Oil was described in GerP 384107 of 1923. Here the oil was slowly distilled with steam or better digested with w at 80–90°, until the Nitroethyl Nitrate it contained was almost completely converted into products sol in w. The undissolved portion was separated and neutralized. It consisted of almost pure NGc. From 100 parts of crude oil about 40 parts of NGc were obtd. The economy of the process depended upon a cheap source of ethylene and upon the possibility of recovering valuable by-products from the destroyed Nitroethyl Nitrate (Ref 7, pp 220–22)

3) *Preparation of NGc from Ethylene Oxide.* Accdg to GerP 376000 of 1920, ethylene oxide (qv) was passed into concd nitric acid, and after saturating the latter with the gas, the concd sulfuric acid was added. The resulting oily substance, separated from spent acid contained at least 16% of N. It consisted mostly of NGc and polymerization products of nitrated ethylene oxide. The crude oil could be stabilized by treatment with steam or hot w, as described in GerP 377268 of 1920, listed in Ref 7, p 223

4) *Preparation of NGc by Method of Messing* (Ref 18b) from ethylene thru chlorohydrin & ethylene oxide:

$$2CH_2{:}CH_2 \xrightarrow{2HOCl} 2Cl.CH_2.CH_2.OH \xrightarrow{Ca(OH)_2} 2\overbrace{CH_2.CH_2.O} + CaCl_2 + 2H_2O$$

After separating the oxide it was converted to ethyleneglycol by heating with water and then nitrated with mixed acid

5) *Preparation of NGc by duPont Method*, in which formaldehyde is reacted with carbon monoxide and water to form glycolic acid: $HCHO + CO + H_2O \rightarrow HO.CH_2.COOH$ and after esterification of the acid with methanol, the resulting methylglycolate was reduced to form methanol & glycol: $HO.CH_2.COOCH_3 + 2H_2 \rightarrow CH_3OH + HO.CH_2.CH_2OH$ After separating glycol it was nitrated by mixed acid

EGDN (NGc) was first commercially introduced in 1926 in the USA by Union Carbide Chemical Corp. Original mixture employed in Dynamites contd 20% NGc & 80% NG, but later mixts contg as much as 80% of NGc were used. This has been brought about by the reduction in price to below the cost of NG

Prepn of EGDN and of other nitrated glycols was described by Aubertein (Ref 17c). The yield of EGDN was 93% of theory

Continuous methods using the same installations as for manuf of NG are used now in Europe. Two of these installations, Schmid-Meissner and Biazzi, are described in Vol 3 of Encycl, pp C502–C504. We also know that continuous method of Schmid-Meissner is installed in Argentina at the Naval Explosives Plant. The same plant employs Bofors Continuous Method for manuf of TNT. Accdg to Ref 24, nearly 100% conversion of Gc to NGc is achieved by mixed acid nitration in liquid sulfur dioxide

Note: Rinkenbach & Aaronsen (Ref A) measured the yield of **Diethylene Glycoldinitrate** as a function of the ratio of sulfuric acid to the amount of water present when nitration was complete, holding the nitric acid in a constant excess. They found that a value of 3.15 gave the maximum yield, about 83%, and reasoned that more concd sulfuric acid dehydrated the DEGc to form poly EGc's which gave lesser yields of dinitrates

Properties and Uses of EGDN (Refs 1, 2, 5, 6, 6a, 7, 7a, 8, 9, 10, 11, 12, 12a, 13, 14, 15, 16, 16a, 17, 17a, 18, 18a, 18b, 18c, 19, 19a, 20, 21 & 23):

Ageing Properties of Explosives Containing NGc. See under Uses of NGc

Brisance by Lead Block Compression (Hess Crusher Test) – 30.0mm, vs 18.5mm for NG and 16mm for TNT (Ref 7, p 157 under Nitroglycerine as an Explosive)

Brisance by Sand Test, detd in mixts with 40% kieselguhr, gave for NGc mixts sl higher results than with those contg NG (Ref 19, p 133)

Chemical Properties. On heating with aq solns of alkalies, gradual soln with saponification, similar to NG, takes place. With alc KOH, NGc reacts violently with formation of K nitrate and glycolate (Ref 7, p 224)

Chemical Stability. See Stability

Chemical Test for Purity is conducted by Nitrometer Method, originated by Lange but modified by duPont Co. Detailed description of the test is given in Vol 1 of Encycl, under "AMMONIUM NITRATE, Gasometric Method", pp A373–A378. N content of NGc should be about 18.42%, vs 18.50 for NG

Critical Temperature – 114–116° (Ref 5, p 1200)

Crusher Test. See under Brisance

Decomposition (or Explosion) Equation: $C_2H_4(ONO_2)_2 \rightarrow 2CO_2 + 2H_2O + N_2$. It is an ideal decompn equation because there is enough oxygen for complete combstn. Corresponding equation for NG is: $4C_3H_5(ONO_2)_3 = 12CO_2 + 10H_2O + 6N_2 + O_2$ Equation given by Kast for NG (Ref 7, p 146, Footnote 21): wherein the formation of NO to a slight extent as an endothermic side reaction is assumed

Detonation Velocity of liquid NGc, detd in glass tubes 1 cm in diam with 1 mm wall thickness 2050m/sec and 7300m/sec at d 1.485. For liq NG detd in glass tube 0.39 inch diam and d 1.6 1600–1900m/sec, whereas in steel tube 1.25 inch diam it was 7700m/sec at d 1.6 (Ref 23, p 233). Curme & Johnston (Ref 19, p 132) give 8266m/sec, vs 8484 for NG, when using No 8 cap and PA booster

Energy Content. Comparing the heat of expln, 1705.3cal/g at C_v and water liquid with that for NG (1595.0cal/g), Naoúm (Ref 7, p 225) stated that the energy content of NGc is ca 107% that of NG

Energy in Meter-Kilograms Developed on Explosion of 1 Kg of Material: 695.7×10^6 for NGc vs 647.8×10^6 for NG (Ref 6, p 298 & Ref 19, p 132)

Explosion Reaction. See Decomposition Equation

Explosion Strength or Power. As regards energy content and high deton velocity, NGc occupies first place by the customary methods

for detg explosive strength. If Lead Block Expansion (Trauzl Test) of 10g NGc with water tamping is 600cc, vs 590cc for NG, the strength of NGc is 110% that of NG, whereas its energy content is 107% of NG (Ref 7, pp 226–27). Blatt (Ref 16) gave 170% of PA

Explosion Temperature: 257° in 5 secs (Ref 23, p 143), vs 222° for NG (Ref 23, p 233). Accdg to Wieland & Sakellarios (Ref 3a and quoted in Ref 5, p 1199) NGc can explode at as low as 215° when rapidly heated, and this temp is listed in Ref 19, p 132 (Comp with Temperature of Explosion)

Flame Temperature – 1350°C (Ref 12b)

Flammability (Inflammability). Accdg to Ref 7, p 225, both NGc and expls contg it ignite when in contact with a flame, and burn with sputtering unless a sudden overheating occurs, in which case there is an expln. This behavior is similar to that of NG. In general NGc expls burn more quietly than do those contg NG, and have less tendency to explode than the latter

Fluidity at 20° – 27.54 at sp gr 1.4833 vs 3.47 for NG at sp gr 1.587 and water 100.0 (Ref 5, p 1498)

Gas Volume Produced on Explosion of 1 Kilogram – 736.93 liters at 0° & 760mm Hg, vs 715.53 for NG (Ref 6, p 298 & Ref 19, p 132)

Gas Volume Produced on Explosion of 1 kg, calcd to temp of expln 4209°, 12099 liters vs 11663 liters for NG at 4177° (Ref 19, p 132)

Gelatinizing Power. NGc gelatinizes NC's much faster than does NG. Gelatinization takes place in a short time even at low temps, while with NG heat must be applied (Ref 7, p 224 & Ref 15, p 226)

Heat of Combustion at C_v – 1763.9cal/g or 268.22kcal/mole, vs 1630.4 & 370.5 for NG (Ref 5, p 1498, Ref 6, p 298 & Ref 19, p 132)

Heat of Combustion at C_p – 1752.5cal/g or 266.48kcal/mole, vs 1622.1 or 368.36 for NG (Ref 5, p 1498 & Ref 19, p 132)

Heat of Explosion at C_v, with water liquid 1705.3cal/g and 1580.9cal/g with water gas (Ref 7, p 225), vs 1589cal/g and 1470, respectively for NG (Ref 7, p 148)

Heat of Formation – 55.58kcal/mole or 365.5 cal/g, vs 81.7 or 359.8 respectively for NG (Ref 5, p 1199). Naoúm (Ref 7, p 225) calcd for NG 67.7kcal/mole; 365.5cal/g listed in Ref 19, p 132

Hygroscopicity. Pure NGc is practically nonhygroscopic, in which property it does not differ from NG. Samples kept in an air space saturated with water-vapor at 26° over a period of 15 days and weighed regularly not only failed to absorb moisture but lost some weight. A sample of pure NG under the same conditions did not appreciably increase or decrease in wt (Ref 5, p 1199)

Impact Sensitiveness or Sensitiveness to Shock. It is less sensitive than NG. Tests made on the small BurMines Apparatus, using 500g weight gave the following values for the minimum fall: NGc 110cm and NG 70 (Ref 5, p 1200). Naoúm (Ref 7, p 225) gives 20–25cm for NGc, vs 8–10cm for NG for deton of samples absorbed on filter paper when using Kast Apparatus with 2kg weight. The same values are given in Blatt (Ref 16) and in addition he gives FI=20% PA. In AMCP 706–177 (Ref 23) are given results of tests with PicArsnApp using 1 pound weight and 20mg samples – 4 inches for NGc (p 143), vs 1 inch for NG (p 233)

Inflammability. See Flammability

Initiation Sensitiveness. NGc is very sensitive to initial impulse, and NG is exploded, both in a liquid condition and as in Dynamite by the weakest common cap, a No 1, even without confinement (Ref 7, p 226 & Ref 16)

Magnetic Rotation at 12.6°: specific gravity 0.6686 & molecular rotation 3.7681 (Ref 5, p 1200 & Ref 19, p 132)

Physiological Properties. See under Toxicity

Power. See under Explosive Strength

Pressure Developed on Explosion of One Kilogram in One Liter – 12498kg/cm^2 for NGc vs 12048 for NG (Ref 6, p 298; Ref 19, p 132)

Reaction on Explosion. See Decomposition (or Explosion) Equation

Sand Test for Brisance. See under Brisance by Sand Test

Sensitiveness to Impact (or Shock). See Impact Sensitiveness

Sensitiveness to Initiation. See Initiation Sensitiveness

Solubilities of NGc. At ordinary temps NGc is readily sol in alc, ether, acetone, chlf, benz, NB and toluene, but only sl sol in CCl_4 and benzine. The solubility relations of NGc are not essentially different from those of NG, aside from its soly in water. The following amounts of NGc are dissolved in 1 liter of w: 6.2g at 15°, 6.8 at 20° and 9.2 at 50°; vs

1.6, 1.8 and 2.5, respectively, for NG (Ref 5, p 1199 and Ref 7, pp 224 & 119). Aubertein (Ref 17c) gives 0.56g in 100g of w at 25°; soly of w in 100g NGc – 0.43g

Solubility Effects. Inasmuch as soly of NGc in w is much higher than that of NG, it is expected that a lower yield will be obtd in the course of manuf. However, the possibility of nitrating and washing at low temps offers a method of decreasing this loss (Ref 6, p 299)

Specific Heat at RT – approx 0.4cal/g for liquid or gelatinized (detd by G. Clift)

Stability by Abel Test at 72°. NGc colors KI paper quickly. However, this should be attributed to the relatively high evaporation rate and slight dissociation of the vapors, and does not represent a low chemical stability (Ref 7, p 226)

Stability at 75°. On long continued heating in a loosely covered weighing dish, NGc showed greater stability than did NG (Ref 7, p 226). On p 135 of Ref 7 is Table 4, which shows that after heating for 12 days at 75° only 4.2% of NGc began to decompose (becoming acidic), vs 28.0% NG. The corresponding values for after 18 days of heating were 35.0% for NGc and 54.0 for NG. Complete decompn took place after 25 days for 71.0% of NGc vs 73.5% of NG

Temperature of Explosion (Not to be confused with Explosion Temperature). Accdg to Rinkenbach (Ref 6, p 298), temp developed on expln of NGc is 4209°C, vs 4177° for NG. Same temps are listed by Curme & Johnston (Ref 19, p 133)

Toxicity. Accdg to Naoúm (Ref 7, p 224), NGc causes headache, just like NG, but on account of its volatility its effects on the workmen are more violent than those of NG. In the opinion of Rinkenbach (Ref 6, p 299 and Ref 5, p 1200) the toxic effects produced by NGc are no worse than those of NG and probably are less prolonged. For more detailed description of NGc toxicity, see Curme & Johnston (Ref 19, p 312), E. Gross, M. Bock & F. Helbrung, ArchExptlPathPharmakol **200**, 271 (1942) and under Ethyleneglycol Dinitrate Liquid Propellant for Use in "ADAM" Munition. The munition is classified. See also Ref 18c

Vacuum Stability. No information

Vapor Pressure. Accdg to Rinkenbach (Ref 6, p 299) vapor pressure detd in 1926 gave 0.0565mm Hg at 22°, which is about 150 times as great as that of NG. Curme & Johnston (Ref 19, p 132) gave at 20° 0.0490mm for NGc, vs 0.00038 for NG and at 50° the values are 0.665 and 0.0081, respectively. Jordan (Ref 19b) gave for NGc: 0.02330mm at 15°, 0.07059 at 25°, 0.2190 at 35°, 0.44250 at 45°, 0.9619 at 55°. Naoúm (Ref 7, p 109) gave for NG: 0.00025mm Hg at 20°, 0.00083 at 30°, 0.0024 at 40°, 0.0072 at 50°, 0.0188 at 60°, 0.043 at 70°, 0.098 at 80° and 0.29mm at 93.3°

Velocity of Detonation. See Detonation Velocity

Viscosity. NGc is considerably less viscous than NG. At RT about 8 times as fluid as NG (Ref 6, p 298). The time taken for 5cc to run out of a 10ml pipet was 4.5sec for water, 5.0sec for NGc & 12.5sec for NG. If viscosity of water is designated as 100 then 111 is for NGc and 278 for NG (Ref 7, p 223). Rinkenbach (Ref 5, p 1498) gave at 20° 0.0363 for NGc vs 0.288 poises for NG. Viscosity detd at PicArsn Lab was 4.2 centipoises at 20°. Curme & Johnston (Ref 19, p 132) gave at 20° 0.0421 poises for NGc, vs 0.360 for NG, while at 50°, the values were 0.0214 & 0.0938, respectively

Volatility. NGc is appreciably volatile even at ordinary temperatures and considerably at elevated temps. Naoúm (Ref 7, p 223) gives the following values representing the loss in weight of a 15g sample in an open dish 65mm diam & 40mm high:

1) At 20° after 24hrs 0.14%; 48 hrs 0.37; 7 days 1.63; 12 days 2.06; 30 days 6.91 and 6 months 47.0%

2) At 35° after 24 hrs 3.6%; 48 hrs 8.1; 7 days 21.7; 12 days 46.6; 20 days 68.5 and 40 days 100% loss

For comparison with NG, the following values are given by Naoúm (Ref 7, p 109): 20g of NG in an open dish of 70mm diam lost in 24 hrs: at 50° 0.2%, at 75° 1.6 and at 100° 10%

Uses of Ethyleneglycol Dinitrate

Although known since 1870 to be an expl, it found no practical application because it was too expensive. It was not until 1904 that a patent (DRP 179789) covering its use as an expl was granted, but it was about 8 years before its actual use in expl compns was recorded by Dautriche (Ref 2). The use of NGc on a large scale, as an antifreeze addition to expls based on NG (Dynamites), took place when inexpensive methods of manuf of glycol

were developed, such as the method of Hough, patented in 1916 (Ref 3). Straight NGc was used in Germany during WWI as replacement for NG which was in short supply (Synthetic method of manuf of glycerin was not invented until later)

Studies at USBurMines by Perrott & Tiffany (Ref 6b) have shown that properties of expls contg mixts of NG with up to 25% NGc did not differ (except fr p) from those contg straight NG. Studies by Coates & Perrott (Ref 7a) of **ageing properties** of Gelatin Dynamites have shown that propulsive strength is unaffected by age by both NGc and NG types, but deton velocities of NGc contg Dynamites have shown better ageing properties than those contg NG alone (quoted from Ref 19, p 133)

Compositions and properties of Dynamites contg NGc are given in Vol 5, under DYNAMITE, pp D1590-R to D1592 (See also Ageing of Dynamites in Vol 1 of Encycl, pp A110-R to A112-L

EGDN was used by Germans during WWII in some proplnts. For example, their *Einheitspulver R61* contd 35.5% of EGDN. Its compn is given in Vol 5 of Encycl, p E17-R

The following plastic compns contg EGDN, patented by WASAG-Chemie AG, Essen (DAS 1148924) are described in Ref 22b: 1) EGDN 38.0, CC (collodion cotton) 0.75, Lackwolle "E33" (in 33% soln of butyl acetate) 0.75, liquid DNT/TNT (probably like Tropföl= Dripoil) 2.0, TNT 2.0, AN 52.3, woodmeal 4.0 and iron oxide 0.2% 2) EGDN 34.7, CC 0.8, Lackwolle "E33" (in 33% soln of butyl acetate) 0.8, DNT with TNT 4.5, AN 49.2, woodmeal 2.0 and iron oxide 8.0%

NOTE: Thru the courtesy of Dr W. Schmacker (German civilian assigned to Picatinny Arsenal under the International Professional Scientists Exchange Program) the following info was obtd from WASAG-Chemie Gmbh, West Germany by letter dated 24 July 1973: **Lackwolle E33** is a pressure-cooked modified collodion cotton of 12.0 to 12.2% N. Its stability is less than 2.5cc NO/g and its viscosity by falling ball method of a 33% soln in 90–100% butyl acetate soln is 160sec or by Hercules method 15–18 centipoises

The following liquid propellant based on EGDN was recently developed in the USA: EGDN 87.1±1.2, NPL(1-Nitro-2-propanol) 12.0±1.0 and 2-NDPhA(2-Nitrodiphenylamine) 0.9±0.1%. Its intended use is in "ADAM", which is one of the classified items of munition

A proposed Specification for this liquid propellant is described after the description of analytical procedures for EGDN

No US Military Specification for EGDN exists

Refs for EGDN:

1) Beil **1**, 469, (244) & [521] 2) H. Dautriche, MP **16**, 72(1911–1912) 3) A. Hough, USP 1206222 & 1206223(1916) & CA **11**, 211(1917) 3a) H. Wieland & E. Sakellarios, Ber **53B**, 203 (1920) 4) H. Oehme, USP 1426313(1922) & CA **16**, 3316(1922) 5) Wm.H. Rinkenbach, IEC **18**, 1195–1200(1926) (Prepn & props of NGc)(23 refs) 6) Wm.H. Rinkenbach, ChemMetalEngrg **34**, 296–99(1927) (Glycol Dinitrate in Dynamite manufacture) 6a) A. Schmid, SS **22**, 273–76(1927) 6b) G.St.J. Perrott & J.E. Tiffany, USBurMines **RI 2935** (1927) 7) Naoúm, NG(1928), 218–27 7a) A.B. Coates & G.St.J. Perrott, USBurMines **RI 2923**(1929) 8) Marshall **3**(1932), p 46 9) Stettbacher (1933), 169–71 10) G. Guastalla & G. Racciu, Industria-Chimica **8**, 843(1933) & CA **27**, 5189(1933) 11) A. Schmidt, SS **29**, 262(1934) 12) Pepin Lehalleur (1935), 195–96 12a) Thorpe **4**(1940), 489–90 12b) A.F. Belyaev, Zh-FizKhim **14**, 1009–25(1941)(Rate of burning of EGDN) 13) A.H. Blatt & F.C. Whitmore, OSRD **1085**(1942), p 35 14) Clift & Fedoroff, Vol **2**(1943), p N-33 15) Davis (1943), 225–26 16) Blatt, OSRD **2014**(1944) 16a) Pérez Ara (1945), 279–81 16b) K.K. Andreev, DoklAkadN **51**, 123–26(1946)(Influence of pressure on burning of NGc) 17) Vivas, Feigenspan & Ladreda, Vol **2** (1946), 37–9 17a) ADL, PureExplCompds, Part **2**(1947), p 225 17b) Stettbacher (1948), p 61 17c) P. Aubertein, MP **30**, 7(1948) & CA **45**, 353(1951)(Prepn and props of EGDN and of other nitrated glycols) 18) ADL Synthesis HE's, First Rept (1949), p 33 18a) R.F. Messing, ChemInd **67**, 41(1950) 18b) Belgrano (1952), 57–9 18c) H. Symanski, Folia Med(Naples) **35**, 741–56 (1952)& CA **47**, 4610(1953)(Severe lesions caused by NGc might cause death) 19) Curme & Johnston (1952), 65–7, 130–34 & 312 (Numerous refs) 19b) T. Jordan,

"Vapor Pressure of Organic Compounds", Interscience, NY (1954), Chap 7 20) Yaremenko & Svetlov (1957), 178–79 21) Giua, Trattato **VI** (1)(1959), pp 124–27 22) CondChemDict (1961) – not listed 22a) V. Öhman et al, Explosivst **1961**, 5 & 95–113 (Stability of acidic NGc system) 22b) H. Joachim & H. Schlüter, Explosivstoffe **1964**, 14 22c) Urbański **2** (1965), 142–49 23) AMCP **706-177**, March 1967, "Properties of Explosives of Military Interest", [formerly PATR **1740** (Rev 1958), by W.R. Tomlinson Jr & O.E. Sheffield], pp 143–45 24) Stamicarbon N.V., NethPAppl 6512732(1967) & CA **67**, 731435(1967) 25) Y. Morikawa, Nature **213**(5078), 841–2(1967)(Toxicology) & CA **66**, 84246(1967) 26) O. Boehm, Explosivst **15**(2), 25–33(1967)(Analysis) & CA **66**, 117538(1967) 27) M.L. Weiss & E.L. Litchfield, USBurMines, RepInvest No 6986, 17pp(1967)(Impact sensitivity, and other compounds) & CA **67**, 83542(1967) 28) E. Camera & D. Pravisani, AnalChem **39**(13), 1645–6(1967)(Analysis) & CA **67**, 111212(1967) 29) D.B. Parihar et al, JChromatogr **31**(2), 551–6(1967)(Analysis) & CA **68**, 97217(1968) 30) W. Pietrulla, Fresenius'ZAnalChem **237**(3), 178–92(1968) (Analysis) & CA **69**, 4078(1968) 31) R.J.M. Fraser & N.C. Paul, JCS, B **1968**, (6), 659–63 (Mass spectrum) & CA **69**, 31418(1968) 32) J.E. Evendijk, Explosivst **1968**, 16(7), 152–4(Analysis) & CA **69**, 98116(1968) 33) M.H. Litchfield, Analyst(London) **1968**, 93(1111), 653–9(Analysis) & CA **70**, 18490 (1969) 33a) Sax, 3rd edit (1968), 747 34) A. Dravnieks & M.J. Salkowski, USP 3430482(1969)(Detection in air) & CA **70**, 89339(1969) 35) T.Matsushita, IndHealth (Kawasaki, Japan) **1969**, 7(1–2), 22–30 (Toxicology) & CA **72**, 82682(1970) 36) J. Vick et al, Explosivst **1970**, 18(2), 42–8(Analysis) & CA **73**, 57623(1970) 37) A. Dravnieks & J. Fischman, USP 3568411(1971)(Detection in air) & CA **74**, 128367(1971)

Addnl Refs for EGDN:
A) W.H. Rinkenbach & H.A. Aaronson, IEC **23**, 160–1(1931)
B) R.G. Vines, Nature **160**, 400–1(1947) (Deton vel and small bubbles) & CA **42**, 369(1948)
C) A.F. Belyaev, JPhysChem(USSR) **22**, 91–101(1948)(bp and heat of vapn) & CA **42**, 5227(1948)
D) L. Médard, MP **31**, 131–43(1949)(Shock sensitivity, many other expls) & CA **46**, 11685(1952)
E) J. Boileau & M. Thomas, MP **33**, 155–7 (1951)(Visc, sp gr, n_D) & CA **47**, 5200(1953)
F) R. Steinberger et al, JACS **77**, 4748–50 (1955)(Burning rate) & CA **50**, 3043(1956)
G) G. Rossmy, Ber **88**, 1969–74(1955)(Infrared spectrum) & CA **50**, 11938(1956)
H) N.A.P. Nilsson, USP 2737522(1956) (Manuf, and other compds) & CA **50**, 6796(1956)
I) K.K. Andreev & B.S. Samsonov, NauchDokladyVyssheiShkoly, KhimKhimTekhnol **1958**, No 2, 229–32 (Effect of pressure on heat sensitivity, also NG) & CA **52**, 17713 (1958)
J) P.V. Öhman et al, ChemZtg **83**, 399–406 (1959)(Manuf condns) & CA **54**, 1851(1960)
K) B.B. Coldwell, Analyst **84**, 665–7(1959) (Detection, other compds) & CA **54**, 17887(1960)
L) HerculesPowderCo, BritP 837044(1960) (Manuf) & CA **54**, 24406(1960)
M) C.D. McKinney Jr, USP 2951866(1960) (Manuf) & CA **55**, 2108(1961)
N) P.V. Öhman et al, Explosivst **9**, 95–113 (1961)(Manuf safety) & CA **56**, 6229(1962)
O) H. Yoshikawa & M. Ishii, BullNatlInstHealth(Kawasaki, Japan) No 7, 1–6(1962) (Toxicology) & CA **58**, 14615(1963)
P) N. Zurlo et al, MedLavoro(Milan) **54**, 166–8(1963)(Analysis) & CA **59**, 9070(1963)
Q) T. Akisada, BunsekiKagaku **12**, 443–6 (1963)(Analysis) & CA **59**, 9235(1963)
R) P. Carmichael & J. Lieben, ArchEnvironHealth **7**(4), 424–39(1963)(Toxicology) & CA **60**, 2245(1964)
S) C. Einert et al, AmIndHygAssocJ **24**(5), 435–47(1963)(Toxicology) & CA **60**, 3413 (1964)
T) K. Hotla & R. Yamada, IntemArchGewerbepatholGewerbehyg **20**, 311–15(1963)(Toxicology) & CA **60**, 8540(1964)
U) G. Krien, Explosivst **11**(10), 207–9 (1963)(Analysis) & CA **61**, 11839(1964)
V) K.R.K. Rao et al, CurrentSci(India) **33**(1), 12–13(1964)(Analysis) & CA **60**, 6691(1964)
W) S.K. Sinha et al, CurrentSci(India) **33**(5), 141–2(1964)(Analysis) & CA **60**, 11835(1964)
X) L. Parmeggiani et al, MedLavoro **55**(1), 23–40(1964)(Toxicology) & CA **61**, 3600(1964)

Y) M. Bami & P. Martini, MedLavoro **55**(5), 326–40(1964)(Toxicology review) & CA **61**, 12536(1964)
Z) F.C. Gibson et al, NASA Accession No N64-23112, Rept No AD 438019, 11pp(1964) (Sensitivity with NG) & CA **62**, 8923(1965)
AA) K. Suwa et al, IndHealth(Kawasaki, Japan) **2**(2), 80–6(1964)(Toxicology) & CA **62**, 12364(1965)
BB) H. Hasegawa et al, IndHealth(Kawasaki, Japan) **2**(2), 129–30(1964)(Toxicology) & CA **62**, 12365(1965)
CC) H. Tsuruta et al, IndHealth(Kawasaki, Japan) **2**(3–4), 149–54(1964)(Toxicology) & CA **63**, 18871(1965)
DD) P. Martini & L. Massari, MedLavoro **56**(1), 62–7(1965)(Toxicology) & CA **63**, 3534(1965)
EE) E. Camera, Explosivst **13**(9), 237–48 (1965)(Analysis) & CA **64**, 1889(1966)
FF) M. Hanova, BratislavLekarskeListy **45-II** (4), 220–4(1965)(Toxicology) & CA **64**, 4148 (1966)
GG) M. Yamakawa, KôgyôKayakuKyôkaishi **26**(4), 236–42(1965)(Toxicology review) & CA **64**, 6394(1966)
HH) P. Lingens et al, GerP 1209034(1966) (Gelation of NGc–NG) & CA **64**, 11022(1966)
II) O. Boehm, Explosivst **15**(5), 97–106(1966) (Analysis) & CA **65**, 8654(1966)
JJ) A.F. Williams et al, Nature **210**(5038), 816–17(1966)(Analysis) & CA **65**, 11221(1966)
KK) H. Hennig, Explosivst **14**(9), 193–6(1966) (Analysis) & CA **66**, 30635(1967)

Ethyleneglycol Dinitrate, Analytical Procedures As Practiced at Trojan-US Powder, Allentown, Pa, 18105.

Accdg to the letter of May 7, 1973 from W.J. Carroll, New Products Manager-Explosives of Trojan-US Powder to O.E. Sheffield of Picatinny Arsenal, Dover, NJ, 07801, the Trojan-US Powder is now a producer of EGDN. This material is shipped as a desensitized solution consisting of EGDN 59.75, 2-Nitrophenylamine 0.25 and acetone 40.00%

The specification of Trojan-US Powder for EGDN (without stabilizer) is:

Assay by Nitrometer	98.0%, min
Acidity as HNO_3	0.01%, max
or	
Alkalinity as Na_2CO_3	0.01%, max
Moisture by Karl Fischer	0.25%, max
KI Test at 82.2°C	8 minutes, min

Test procedures (not specifications) are essentially those of MIL-N-246B, 19 Feb 1962, covering Nitroglycerin with the modifications described under 4.3 TEST METHODS AND PROCEDURES which follow

4.3 *TEST METHODS AND PROCEDURES*

4.3.1 *Determination of Moisture in EGDN by Karl Fischer.* Weigh accurately a 5–10g portion of the sample (sample size shall be in accordance with the strength of the Karl Fischer reagent) of the EGDN into a tared narrow-necked stoppered flask. Titrate directly until a brown tinge persists in the soln for 30 seconds (Potentiometric endpoint indicators may also be used in titration with Karl Fischer reagent). Calculate as follows:

$$\text{Percentage moisture} = \frac{100(KF)}{W}$$

where: K = ml of Karl Fischer reagent used in titration
F = Reagent factor (g of water per ml of reagent)
W = Weight of sample in grams

Note: A detailed description of Karl Fischer Method is given in Vol 5 of Encycl, pp D1622 to D1627. It is practically the same as Method 101.4 of MIL-STD-650

4.3.2 *Determination of Acidity or Alkalinity.* By means of a pipette, transfer a portion of approx 10g of the sample to a tared 250-ml beaker, reweigh and dissolve in 100ml of benzene. Move the soln to a 250-ml separatory funnel and shake the contents of it with a 50-ml portion of neutral distilled water. After separation of layers run the water layer into the above 250-ml beaker. Repeat the operation using another 50ml of distd w and transfer the bottom layer to the same beaker. Add to beaker several drops of bromomethyl blue indicator. If the soln turns yellow titrate immediately with standard 0.01N NaOH soln and if blue titrate immediately with standard 0.01N sulfuric acid. The change of color is taken as end point. Run a blank detn on 100ml of benzene alone and correct the volume alkali or acid required for titration. Calculate the percentage of acidity as nitric acid or alkalinity as Na carbonate as follows:

$$\text{Percentage Nitric Acid} = \frac{6.3(V-v)N}{W}$$

where: V = ml NaOH soln required for sample
v = ml NaOH soln required for blank

N = Normality of NaOH soln
W = Weight of sample in grams

$$\text{Percentage of Na carbonate} = \frac{5.3(V-v)N}{W}$$

where: V = ml sulfuric acid soln required for sample
v = ml sulfuric acid soln required for blank
N = Normality of sulfuric acid soln
W = Weight of sample

4.3.3 *Determination of Nitrogen* (Assay by Nitrometer).

Accdg to W.J. Carroll, EGDN is dried at Trojan laboratory by desiccation prior to nitrogen determination, thus eliminating the filtering steps described in 4.3.3.2 and 4.3.3.3 of MIL-N-246B. Since moisture content of the resulting sample is less than 0.03%, no correction is made for this negligible amount, and the calculation thus becomes:

$$\text{Percentage of Nitrogen} = A/W$$

where: A = Reading of the mercury level on the measuring tube
W = Weight of sample in grams

4.3.3.1 *Standardization of Nitrometer.* Standardization of the DuPont 5-part form of Lunge's nitrometer, or equal, shall be made by Method 209.3 of Military Standard MIL-STD-286

Note: A detailed description of standardization and tests with nitrometer are given in Vol 1 of Encycl, pp A373 to A376 with a drawing of nitrometer on p A374-L

4.3.3.2 Not used for EGDN

4.3.3.3 *Procedure.* Eliminate the part dealing with filtering thru filter paper and start as follows:

Transfer from a moisture-proof receptacle contg EGDN dried in desiccator, 0.70 to 0.75g sample (contg less than 0.03% moisture) to a tared 25-ml beaker and weigh accurately immediately. Add 5ml of glacial acetic acid and stir the mixture with a small glass rod until the EGDN has dissolved. With the aid of this rod, transfer the soln quickly to the cup of the generating bulb, making certain that no air is drawn into the generating bulb. Measure 25ml of sulfuric acid (94.5±0.5%) into a graduate. Then rinse the beaker and glass rod by means of successive 5-ml portions of this acid, transferring each portion of the rinsing to the generating bulb cup. Follow this by stirring the acid in the cup with glass rod and draw the rinsing into the bulb. Any air drawn into the bulb shall be forced out and the upper stopcock closed when the sample has been completely drawn into the generating bulb

Caution: The lower stopcock must remain open and the mercury reservoir adjusted so that the level of the mercury in the reservoir is 13 to 15 inches below the level of the mercury in the generating bulb. The generating bulb shall be wrapped in a strong towel to prevent injury to hands and body, on accidental breaking of bulb, by glass splinters and acid during shaking operation

With the lower stopcock open and upper stopcock closed, shake gently the generating bulb while keeping its lower end in a fixed position until most of the gas has been generated. Adjust the mercury reservoir so that the mercury in the generating bulb drops nearly to the lower shoulder, and then close the lower stopcock. Shake vigorously for 3 minutes, place the bulb on the rack and allow to stand for 3 minutes. Remove the bulb from the rack, shake for additional 3 minutes and replace on the rack for 3 minutes. After repeating the above procedure once more, transfer the generated gas thru upper stopcock to the measuring and compensating tubes adjusted until the level in compensating tube is approx at the standardization mark. A levelling device shall be used which will make possible accurate adjustment of the levels. Note the reading on the measuring tube and calculate the percentage of nitrogen of the sample by the equation indicated above. It should be not lower than 98% of 18.42%

4.3.3.4 *Determination of Stability by KI Test.* Filter a portion of EGDN sample thru two thicknesses "S and S" Number 604 filter paper or equivalent. Transfer a 2ml portion of filtered material, by means of a pipette, to each of three test tubes which are 5.5 inches long, 0.50 inch internal diameter and 0.62 inch outside diameter (Care should be taken during transfer not to leave droplets of the EGDN on the sides of the test tubes). Stopper each tube by means of a new tightly fitting cork thru which passes a tightly fitted glass rod equipped with a platinum hook for a strip of standard KI-starch indicator paper, about

1 inch long and 3/8 inch wide. Dip a small diameter glass rod into a 50% soln (by volume) of pure glycerin in distilled water, and as the rod is withdrawn, contact is made with the sides of container so as to minimize the amt of liquid adhering to the rod. Draw the moistened rod across the upper part of paper so as to produce a level and distinct line of demarcation on lower edge of the wet upper area. Prepare a blank by suspending a moistened strip of standard KI-Starch test paper in a clean dry test tube

Adjust the temperature of the heat tube bath to 82.2±1°C and insert all four test tubes into the heating soln. The depth of immersion should be approx 2 inches. Place the bath in such a position that the test tubes are viewed against a white background illuminated by bright diffused daylight. Note the time of insertion of the tubes into the bath. During the test the line of demarcation on the test paper strip should be 3 inches above the EGDN level in the test tube. The line of demarcation in the blank tube is regulated at an equivalent height of the tube. Consider the end point of test to be the first appearance of any discoloration at the line of demarcation between the wet and the dry portion of the test paper in the sample tube. This discoloration should be in excess of discoloration observed at the same position on the test paper in the blank. Note the time for completion of the test to the nearest minute. Consider the minimum time for the three tubes to represent the heat test value of the sample

After the test, rinse all rods and tubes with acetone to remove EGDN, followed with warm soapy water and then with large amt of tap water and finally with distd w. Dry everything in an oven at approx 80°C

Determination of EGDN in Mixtures with NG by Means of the Refractometer. At the time (1927) of investigation by Mr Rinkenbach of properties of EGDN, there was no accurate method for its detection in mixtures with NG. The cryoscopic method developed in 1920 by M. Marqueyrol & E. Goutal, Bull(Fr) **27**, 443, was in the opinion of Rinkenbach, while fairly accurate for mixtures of the pure substances, yielded very erroneous results when applied to mixtures extracted from commercial expls. This was attributed to the impossibility of entirely removing oils, fats and resins by freezing as recommended by M&G

As the difference between refractive indices of NG and EGDN is fairly large, Rinkenbach decided to apply this difference to analysis of EGDN–NG mixtures. He used a precision Leitz refractometer, equipped with a water jacket and calibrated thermometer. Indices were detd over a wide range of temps for each substance previously thoroughly purified as indicated in Refs 1, 2 & 3, and the curve was drawn which in each case proved to be a straight line. The values, accurate to the 4th decimal place, could be read at different temps from this curve and they were tabulated (See Table Et 5)

Table Et 5 – Refractive Indices of Pure Compounds

°C Temperature	Nitroglycerin	Ethyleneglycol Dinitrate	Diethyleneglycol Dinitrate	70% Nitroglycer 30% Nitropolyglycerin
15.0	1.4751	1.4491	1.4536	1.4767
20.0	1.4732	1.4472	1.4517	1.4748
25.0	1.4713	1.4454	1.4498	1.4728
30.0	1.4693	1.4435	1.4479	1.4709

Inspection shows that the refractive indices of nitrates of the glycols are practically identical and much lower than those of Nitroglycerin and the Nitroglycerin-Nitropolyglycerin mixture. Determination of the refractive index of a sample should give at least a qualitative indication of the presence of the glycol compounds

Tests with Mixtures:

In order to make this method quantitative mixtures of Nitroglycerin and Ethyleneglycol Dinitrate were made up from accurately weighed portions of each and studied in the same manner as were the pure compounds. The curves derived gave the values in Table Et6

Concentration-refractive index curves at 15°, 20°, and 25°C. were plotted from these values. It was found that the points were uniformly below a straight line joining the values for the pure compounds at the given temperature. From the average curve drawn from each temperature the values in Table Et7 were read off

Table Et6 – Refractive Indices of Mixtures of Ethyleneglycol Dinitrate and Nitroglycerin

Percent EGDN	17.35	33.71	49.17	61.93	79.23
Percent NG	82.65	66.29	50.83	38.07	20.77
n_D^{15}	1.4704	1.4651	1.4617	1.4581	1.4539
n_D^{20}	1.4685	1.4634	1.4597	1.4562	1.4519
n_D^{25}	1.4666	1.4617	1.4577	1.4543	1.4499
n_D^{30}	1.4646	1.4600	1.4556	1.4524	1.4579

Table Et7 – Effect of Varying Proportions on Refractive Indices of Mixtures of Ethyleneglycol Dinitrate and Nitroglycerin

Ethylene-glycol Dinitrate Percent	Nitro-glycerin Percent	n_D^{15}	n_D^{20}	n_D^{25}
0	100	1.4751	1.4732	1.4713
10	90	1.4722	1.4703	1.4684
20	80	1.4694	1.4675	1.4656
30	70	1.4666	1.4648	1.4628
40	60	1.4640	1.4621	1.4601
50	50	1.4613	1.4595	1.4575
60	40	1.4587	1.4569	1.4548
70	30	1.4562	1.4544	1.4523
80	20	1.4537	1.4520	1.4498
90	10	1.4513	1.4496	1.4476
100	0	1.4491	1.4472	1.4454

These results show a maximum deviation of -3 per cent to ethyleneglycol dinitrate, or 0.0008 in refractive index from the straight line connecting the 100 per cent values. They also represent a maximum deviation of ± 2 per cent, or 0.0005 from any of the points by which the curves were determined. The values at 15° C came the nearest to giving a smooth curve passing through all points, and this may be considered the optimum temperature at which to work

The foregoing gives a method by which the proportions of the gylcol compounds in admixture with Nitroglycerin or Nitroglycerin and Nitropolyclycerin may be determined with a fair degres of accuracy when the pure components only are present. As such it is particularly applicable to the analysis of nitrated mixtures in plant operation

Effect of Impurities:

The effect of the presence of a small amount of oily impurity, such as is found in ether extracts of Dynamite, was next investigated. A Dynamite known to contain Nitroglycerin but no Nitropolyglycerin or Ethyleneglycol Dinitrate was extracted with ether in the usual manner. The extract was evaporated until all ether had been driven off under low pressure and a residue consisting of Nitroglycerin and separated oils, fats, and resins were obtained The Nitroglycerin was separated by filtration at 30° C and studied by means of the refractometer. It was found that if the temperature was lowered to below 20° C more oils, fats, and resins separated and obscured the field. The Nitroglycerin was then chilled to 0° C, the precipitated oils, fats, and resins were filtered off, and the clear filtrate was studied under the refractometer. The two series of values were used to construct curves from which values at regular temperature intervals were read off (Table Et8) For purposes of comparison corresponding values for pure Nitroglycerin are given

Table Et8 – Refractive Indices of Pure and Dynamite Nitroglycerin

Temp. °C	Pure NG	Dynamite NG Before Freezing	Dynamite NG After Freezing
15.0	1.4751		1.4838
20.0	1.4732	1.4819	1.4820
25.0	1.4713	1.4804	1.4802
30.0	1.4693	1.4781	1.4783

From this table it is evident that the application of the curves derived for mixtures of the pure compounds to the interpretation of values obtained on Dynamite extractives would give results indicating a proportion of Nitroglycerin much higher than that actually present. It is also apparent that the oils, fats, and resins present in Dynamite extractives cannot be removed entirely by freezing, as was recommended by Marqueyrol & Goutel

Application of Method

Inasmuch as the oils naturally present in Dynamite "dopes" vary considerably and the types of greases used for graining purposes also vary to some extent, it is improbable that a satisfactory correction for the error caused by the presence of the substences could be detd and applied to a Dynamite extractive

Accordingly, the application of the method and data given in the case of commercial expls, must be restricted to the qualitative detection of EGDN (and of DEGDN). To the analysis of mixtures that have not dissolved oils and fats from the other constituents of Dynamite, the method can be applied quantitatively, and so is of value in factory-control work

Refs: 1)Wm H.Rinkenbach, IEC **18**, 1195 (1926) 2) Ibid, **19**, 925 (1927), 3) Ibid, **19**, 1291-94 (1927)

DETERMINATION of EGDN in MIXTURES WITH NG by INFRARED METHOD. EGDN is often used in admixture with NG in expls, as the mixture possesses the generally desirable characteristic of having a lower freezing point than either NG or EGDN alone. As precise methods for the analysis of such mixts are lacking a method was developed at Picatinny Arsenal (Ref 5) using infrared spectroscopy

The method involves making absorbance measurements of 20% soln of the sample in benzene at the peak of the 9.20 micron band for NG and of a 5% soln of the sample in 2-nitropropane at the peak of the 9.60 micron band for EGDN. The measured absorbances, after correction for solvent and cell absorbance, are used to calculate percentages of NG and EGDN by the method of "Successive approximations", described in Ref 5 and here

Note: Benzene was selected as a suitable solvent because of its small absorbance at the point of measurement for NG; 2-nitropropane was selected as a suitable solvent because of its small absorbance at the point of measurement for EGDN. In addition, the two solvents were selected because they readily dissolve both NG and EGDN. Both solvents were pretreated with andydrous Na sulfate and NaCl to minimize the tendency to dissolve NaCl cell

Definition of Terms Used:

Transmittance (T): Ratio of radiant power transmitted by sample to radiant power incident to it

Absorbance (A); Logarithm to the base 10 of the reciprocal of the transmittance

Absorptivity (a): Ratio of the absorbance to the product of the concentration and the length of the optical path. (If only one cell is used for all the work, the path length will be constant and can be left out of all calcns without introducing an error. To simplify the calcn, all measurements were made with the same 0.209mm cell. Hence, all the absorptivities were referred to a 0.209mm basis)

Apparatus: A Beckman, Single Beam Infrared Spectrophotometer, Model IR 2, with rock salt (NaCl) optics was used in accordance with the instruction manual for the instrument. All absorbance measurements were made at the peaks of the bands, using a single NaCl cell of about 0.2mm thicknss previously calibrated by the interference fringe method described in Ref 1 and found to be 0.209mm in thickness. Condition of measurement are shown in Table E9

TABLE E9

	NG	EGDN
Wavelength, microns	9.2	9.6
Slit,mm	0.29	0.78
Gain	10	10
Period Seconds	2	2
Shutter	Glass	Glass

Determination of Cell Absorbance and Absorptivity of NG, EGDN, Benzene and 2-Nitropropane. By means of a 0.2mm cell filled with a soln of 15% NG in benz, the peak of a 9.20-micron band was located by very slowly scanning from about 0.05μ below to about 0.05μ above 9.20μ. A 0.2mm cell filled with a soln of 5% EGDN in 2-nitropropane was similarly used to locate the peak of the 9.60-micron band. This procedure was followed to insure measurement of the maximum absorbanc of the two bands

Two cells, one of 0.2mm and another of 0.4mm were calibrated by the method described in Ref 4 and found to be 0.209mm and 0.419mm, respectively. These cells were filled with benz and the total absorbance of each cell was meas-

ured at 9.20 microns. Then the cells were rinsed with 2-nitropropane, filled with it and measured at 9.60 microns. This gave:

Absorptivity of benzene (a)=

$$\frac{(A_{0.419}-A_{0.209})\ 0.209}{0.419-0.209}$$

Absorptivity of 2-nitropropane (a)=

$$\frac{(A'_{0.419}-A'_{0.209})\ 0.209}{0.419-0.209}$$

Cell absorbance of 0.209mm cell = $A_{0.209}$-a, where $A_{0.419}$ and $A_{0.209}$ are, respectively, the absorbance of the 0.4 and 0.2mm cells filled with benz at 9.20μ and $A'_{0.419}$ and $A'_{0.209}$ absorbances of 0.4 and 0.2mm cells filled with 2-nitropropane at 9.60μ. The values obtd are shown in Table E10

TABLE E10

	Wavelength 9.20μ	9.60μ
Absorptivity of benzene (Absorbance per 0.209mm)	0.129	—
Absorptivity of 2-nitropropane (Absorbance per 0.209mm)	—	0.160
Absorbance of 0.209mm cell	0.062	0.061

The absorptivities of NG and EGDN were detd at points corresponding to the maximum absorption of the measured bands. This was done by measuring the absorbance of known sols ranging in concns from 0.5% to 20%. The absorptivities of NG and EGDN were calcd for each soln by applying the following equations:

$$\text{Absorptivity}=\frac{A_{total}-A_{cell}-A_{solvent},}{\text{Percent of material in soln, expressed as a decimal}}$$

where:

A_{total} = Total absorbance of cell filled with soln of solvent and ingredient

A_{cell} = Absorbance of cell at the particular wavelength

$A_{solvent}$ = Absorbance of the solvent at the particular wave length (calcd by multiplying the absorbtivity as listed above, by the percent of solvent in soln). Thus, a 10% benz soln of NG corresponds to 90% benz)

The absorptivities of NG & EGDN obtd in this investigation are listed in Table 1 of Ref 5, reproduced here as Table E11

TABLE E11
Absorptivities of NG and EGDN
(Absorbance per 0.209mm)

Ingredient	Percent Present	Absorptivity (a) at Wavelengths: 9.20μ	9.60μ
NG	0.5	4.00	4.60
	1.5	4.23	4.27
	5.0	4.32	4.30
	10.0	4.35	4.31
	20.0	4.58	4.28
	average	4.29	4.35
EGDN	0.5	0.80	15.60
	1.5	0.87	15.13
	5.0	0.90	15.08
	10.0	0.98	14.66
	20.0	0.92	14.68
	average	0.89	15.03

Determination of Ingredients:

A 5% soln of the sample in 2-nitropropane and 20% soln of the sample in benzene were prepd for the analysis. If, when using these solns, a measurement fell outside of absorbance values 0.19–0.70 (65% - 20% transmittance), an addnl soln was used to bring the readings within the limits. (Optimum absorbance values are around 0.43 or 37% transmittance). With the 0.209mm cell, the absorbance of the benz soln was measured at the peak of the 9.20μ band and the absorbance of 2-nitropropane soln at the peak of the 9.60μ band. The percentages of the components were calcd by the method of "successive approximations"

The 1st approximation for NG was as follows:

$$\%NG = \frac{(A_{total} - A_{cell} - A_{solvent})\ 100}{a}$$

where:

A_{total} = Absorbance at 9.20μ of cell filled with benz soln of sample

A_{cell} = Absorbance of cell at 9.20μ

$A_{solvent}$ = Absorbance of benzene at 9.20μ (This value is obtd by multiplying the absorptivity of benz at 9.20μ by the % of benz in soln, that is, a 20% soln of sample will contain 80% benzene)

a = Absorptivity of NG at 9.20μ (average value 4.29 from Table 1)

Note: Though the bands to be measured in the detn of each component are selected so as to keep interference from the other constituent at a minimum, there was some absorbance at each band by the component other than the one for which the measurement was made. Correction had to be made for this interfering absorbance. Since no knowledge as to the amt of each constituent was available when the first approximation of NG was calcd, no accurate correction could be made for EGDN. No such correction was attempted and, as a result, the value obtd for NG was only approximate (1st approximation). In calcg for NG, the values obtd were taken as 1/5th of the total value since absorbance measurements were made on 20% solns

The 2nd approximation for NG was calcd as follows:

$$\%NG = \frac{(A_{total} - A_{cell} - A_{solvent} + A_{EGDN})100}{a}$$

where:

A_{total} = Absorbance at 9.20μ of cell filled with the benz soln of sample

A_{cell} = Absorbance of cell at 9.20μ

$A_{solvent}$ = Absorbance of benz at 9.20μ (This value was obtd by multiplying the absorptivity of benz at 9.20μ by the %-age of benz in soln, that is, a 20% soln of the sample was considered to contain 80% benz)

A_{EGDN} = Absorbance of 9.20μ due to EGDN obtd as in the following example: Assuming that the %age of NG found in the preceding approximation for NG was 15% in the 20% benz soln, the original mixt contd 15/20th or 75% NG and 25% EGDN. Therefore, in this case, EGDN = (0.25 x 0.20) x 0.89 (Ave from Table 1) = 0.0445

a = Absorptivity of NG at 9.20μ (Ave value = 4.29 from Table 1)

When the 2nd approximation for NG had been obtd, the procedure for approximation was repeated until the values obtd for NG became constant. These values were multiplied by 5 to obtain the total value for NG in the NG-EGDN mixture

The 1st approximation for EGDN is as follows:

$$\%EGDN = \frac{A'_{total} - A'_{cell} - A'_{solvent} - A'_{NG})100}{a}$$

where:

A'_{total} = Absorbance at 9.60mμ of cell filled with soln of sample in 2-nitropropane

A'_{cell} = Absorbance of cell at 9.60μ

$A'_{solvent}$ = Absorbance of 2-nitropropane at 9.6μ. (This value was obtd by multiplying the absorptivity of 2-nitropropane at 9.6μ by the %-age of 2-nitropropane in soln, that is a 5% soln of the sample was considered to contain 95% of 2-nitropropane)

A'_{NG} = Absorbance at 9.6μ due to NG, obtd as in the following examples: Assuming that the %-age of NG found in the calcn for NG was 15% in the 20% benz soln (or 75% NG in the sample), then for a 5% of 2-nitropropane soln, $A'_{NG} = (0.75 \times 0.05) \times 4.35$ (Ave from Table 1) = 0.1631

a = Absorptivity of EGDN at 9.60μ (Ave value 15.03 from Table 1)

In calcg for EGDN, the values obtd were 1/20th of the total value since absorbance measurements were made on 5% solns. When the 1st approximation for EGDN had been obtd, the procedure of approximations were repeated until the values obtd for EGDN became constant. This constant value was multiplied by 20 to obtain the total value for EGDN

In Table 2 of Ref 5, p9 (not reproduced here) are listed values obtd for eight synthetic mixts of NG & EGDN. Three trials were run for each mixture Average absolute error (ave found minus ave added) was calcd for NG as 0.18% and for EGDN as 0.14%. Average relative error $\left(\frac{\text{Absol error x 100}}{\text{Average added}}\right)$ was found for NG 1.7% and for EGDN 1.8%

For employing an extended examination of explosive oils, it is necessary, accdg to Pierson (Ref 6, pp 1355 & 1362) to obtain about 10g of filtered and dried expl oil. The oil can be obtd by extracting with ether a suitable amt of Dynamite placed in a 3-inch Büchner funnel, using a rapid filtering paper. This method is described in Ref 6, p 1355 and in Ref 7, pp D1642-R & D1643-L. If qualitative analysis of this oil indicates the presence of aromatic nitrocompds or of ingredients like DEGDN or Nitrosugar, they must be removed from the expl oil prior to determinations of NG & EGDN. Separation may be accomplished by a chromatographic method (Ref 6, p 1363).

When it is decided that nothing else but NG and NGc remain in the expl oil, it is advisable to determine the amount of NG by Hercules Method D90-3e using periodic acid. This method is described in Ref 7, p D1640-R & D1641. If periodic acid analysis gives less than 90-100% NG, the presence of EGDN is indicated and it can be tentatively estimated as equal to 100-%NG. After this the IR method can be employed

Refs: 1) D.C. Smith & E.C. Miller, JOpt Soc of America **24**, 130-34 (1944)(Cell Calibration)
2) Bull of Society of Applied Spectroscopy **4**, (2), Jan 1949 (Nomenclature)
3) D.Z. Robinson AnalChem **24**, 619 (1952)(Qual Analysis with IR spectrophotometers)
4) F.Pristera, PATR **1821**,& Applied Spectroscopy, **7**, 115(1953)(IR method)
5) W.Huff, M.Halik & F. Pristera, PATR **2472**(1957) (IR analysis of NG-EGDN mixtures)
6) R.H. Pierson in Std Methods Chem Analysis **2B** (1963), 1350-64 (Dynamites)
7(Fedoroff & Sheffield, PATR **2700, Vol 5**, (1972), D1639 to D1643

Ethyleneglycol Dinitrate Liquid Propellant for Use in the "ADAM" Munition ("ADAM" is classified item). Composition of propellant: EGDN 87.1 ±1.2, 1-NPL (1-Nitro-2-propanol) 20.0 ±1.0 and 2-DNPA (2-Nitrodiphenylamine) 0.9 ±0.1%

1.0 Scope. This document covers the requirements and tests for Liquid Proplnt of above compn

1.1 Safety. The proplnt in bulk form is a secondary HE and must be handled according to safety rules

1.2 Toxicity. The bulk proplnt and the EGDN may cause dilation of blood vessels, headaches, nausea, cyanosis, and reduced blood pressure. Extreme exposures resulting from inhalation, skin contact or ingestion may result in central nervous system depression, coma, and respiratory paralysis. Drinking of any quantity of the liquids must be avoided. Alcoholic beverages aggravate the health hazard. Severe headaches after exposure may diminish after development of tolerance. Ventillation should be used to reduce the inhalation hazard. Protective gloves and aprons are helpful in avoiding skin contact and frequent washing with soap and water will minimize skin absorption. Alcohol or other solvents should not be used on the skin. The threshold limit value ceiling is 0.2ppm. No inhalation or skin irritation hazard exists with 1-NPL

2.0 Applicable Documents:

MIL-STD-109: Inspection, Terms and Definitions
MIL-N-3399B: 2-Nitrodiphenylamine
There are no Specs for Ethyleneglycol Dinitrate

Requirements and Tests for the Above EGDN Liquid Propellant described in Proposed Specification (Code Ident No 19203), attached to the letter of October 5,1972 of Mr. D.E. Seeger (Chief, Explosives Application Section ADED,) to Mr. Frank Pristera (Chief, Instr Anal Res Sect, FRL), both at Picatinny Arsenal, Dover, NJ

3.0 Requirements

3.1. Product Acceptance. Propellant furnished accdg to this document shall be inspected for quality in accordance with qualification and acceptance inspection requirements, outlined under Quality Assurance Provisions, items 4.1, 4.1.1, 4.1.2 and 4.1.3 (not included in our description)

3.2 1-Nitro-2-propanol (NPL). Suggested source: (Commercial Solvents Corp, Terre Haute, Indiana

3.2.1 Chemical Composition of NPL. When tested as in 4.3.1, the result shall match the typical IR curve presented in Fig Et16. The presence of additional absorption peaks shall require rejection if the indicative contamination is judged excessive by the analyst

Note: Rejection criteria are not given

3.2.2 Purity of NPL. The sum of the areas of the gas chromatographic peaks of the impurities, with the exception of the acetone peak, shall not exceed 6% of the area of the peak for NPL, when tested as specified in 4.3.2

3.2.3. Moisture Content of NPL, shall not exceed 0.5%, when tested in accordance with 4.3.3

3.2.4. Ignition or Explosion Temperature of NPL shall be 420 ±30°, when tested in accordance with 4.3.4

3.2.5. Thermal Stability of NPL shall not be lower

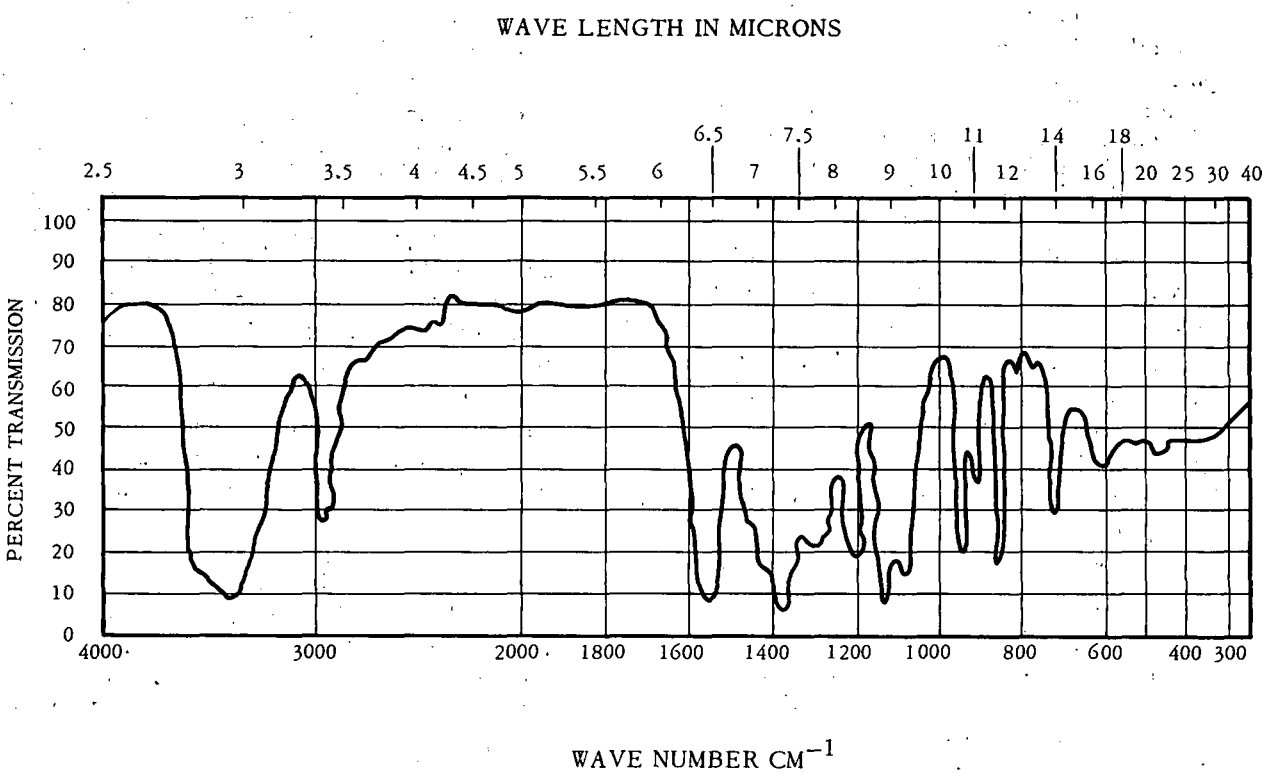


FIGURE Et16 INFRA-RED CURVE FOR 1-NITRO-2-PROPANOL

than 8 minutes, when tested in accordance with 4.3.5

3.3 Ethyleneglycol Dinitrate (EGDN). See the procedure developed at the Trojan-U.S. Powder Company, Allentown, Pennsylvania, 18105, forwarded to us by Mr. W. J. Carrol described here on pp E266-68

3.3.1. Chemical Composition. The EGDN contg a minimum of 0.25% 2-NDPA shall be tested per 4. 3.1. The result shall match the IR Standard presented in Fig Et 17. The presence of additional absorption peaks shall require rejection if the contamination is judged excessive by the analyst (See Remark 1 at the end of this item)

3.3.2. Purity of EGDN. The area under the gas chromatographic peak for acetone shall be less than 0.6% of the area for EGDN. The sum of the areas of all other impurity gas chromatographic peaks shall not exceed 0.05% of the area of the peak of EGDN. Test as specified in 4.3.2

3.3.3. Moisture Content of EGDN shall not exceed 0.25%. Test as specified in 4.3.3

3.3.4. Ignition or Explosion Temperature. The EGDN when tested as in 4.3.4, shall ignite or explode at 217 ± 2°

3.3.5 Thermal Stability. When the EGDN is tested as specified in 4.3.5, the result shall be not lower than 8 minutes

3.4. The 2-Nitrodiphenylamine used in the Liquid Propellant shall meet the requirements of Specification MIL-N-3399B, which is included in Vol 5 of Encycl, p D1428-L, under DIPHENYLAMINE AND DERIVATIVES

Note: Accdg to N.M.Liszt, the bromination procedure should be 10 minutes, instead of 1 minute

3.5 Finished Propellant

3.5.1. Propellant Formulation is given at the beginning of "Ethyleneglycol Dinitrate Liquid Propellar

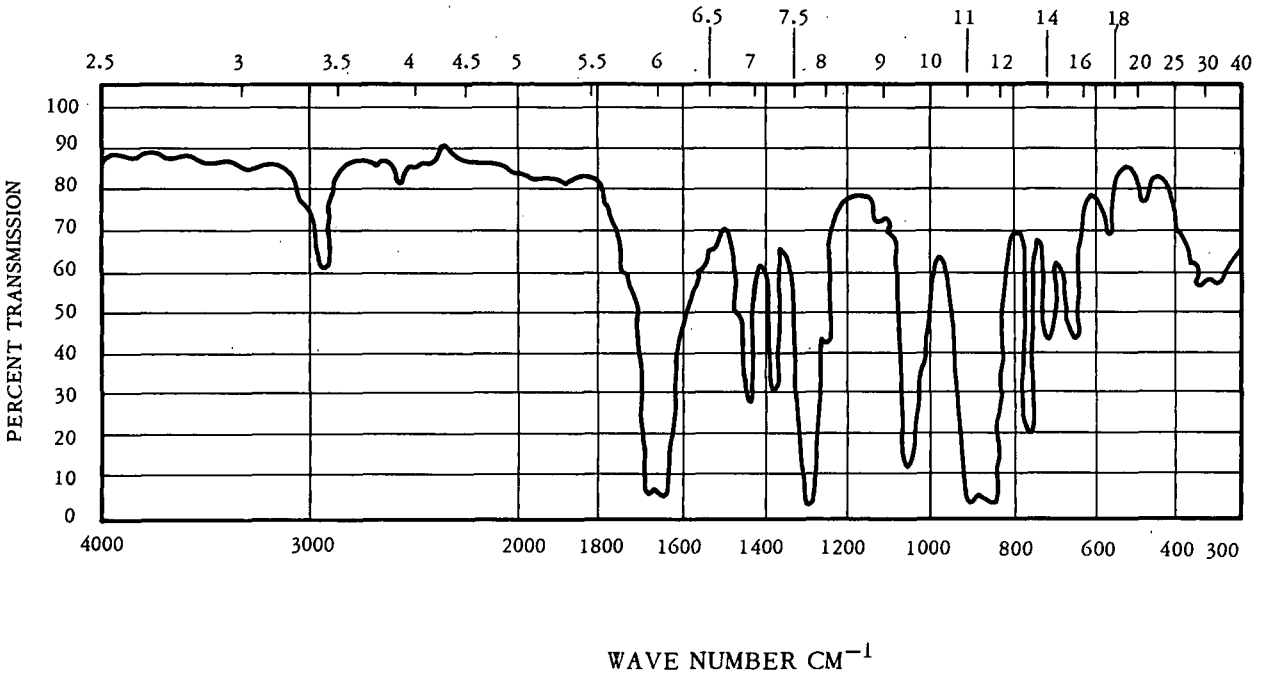


FIGURE Et17 INFRA-RED CURVE FOR ETHYLENE GLYCOL DINITRATE WITH 0.25% 2-NDPA

for Use in the ADAM Munition". The mixture shall be analyzed by the procedure outlined in 4.3.2. The ratio of the area under the gas chromatographic peak for EGDN shall equal 1.32 ± 0.12. The 2-NDPA shall be analyzed by the procedure outlined in 4.36

4.0 Quality Assurance Provisions

4.1. General Quality Assurance Provisions

4.1.1. Contractor Qyality Assurance System

4. 1.2. Submission of Product

4.1.3. Government Verification

4.1.3.1. Surveillance

4.1.3.2. Product Inspection

4.2. Inspection Provisi on

4.2.1. Lot Formation

4.2.2. Examinati on

Paragraphs 4.0 thru 4.2.2 above not included in our description of Proposed Specification

4.3. TEST METHODS AND PROCEDURES

4.3.1. Chemical Analysis by Infrared (IR) Method

Chemical identity of the EGDN and NPL used in this proplnt and presence of substantial amount of other substances shall be checked by making an IR analysis, using a thin film of sample betn KBr windows and comparing these analyses with the appropriate IR absorption curves shown in Figs Et16 & Et17. A typical IR spectrum of finished propellant is shown in Fig Et18

4.3.2. Purity of EGDN, NPL, and the Ratio of Mixtures of these Two Components is suggested to be tested by Gas Chromatographic Analysis, such as described in Vol 3 of Encycl, pp C293-R & C294-L, under CHROMATOGRAPHY

The following conditions shall be used for this analysis:

Column: 5% carbowax 20M on chromasorb 6 (6ft x 1/8 in) column

Carrier gas: Helium at 30 milliliters per minute

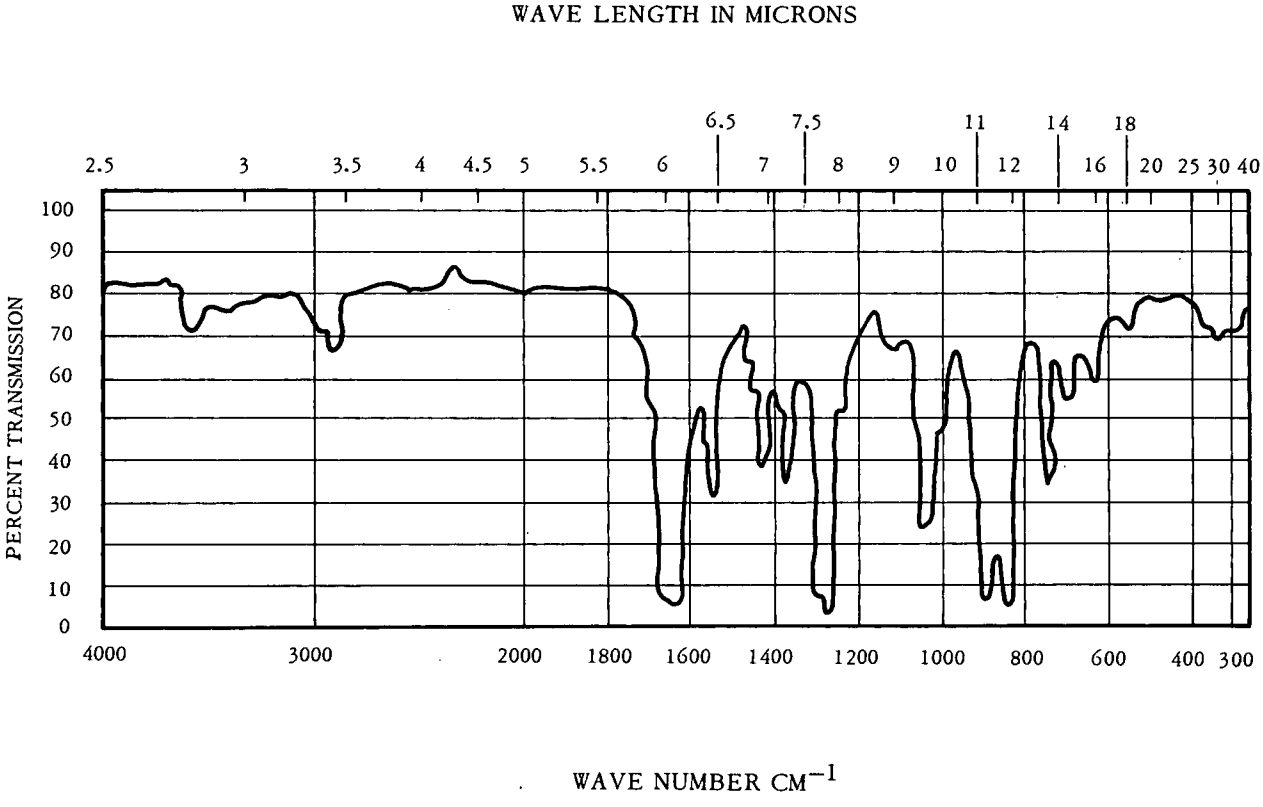


FIGURE E-18 IR CURVE FOR PROPELLANT

Injection port: 150°C
Detector: Flame ionization
Detector oven: 300°C
Oven Temperature: 130° C isothermal
Time per run: 30 minutes

An electronic integrator is used to measure areas and retention times. Using a 1 microliter syringe, introduce a 0.5 microliter sample and start the integrator. Under these conditions, acetone will elute at 0.5 to 0.6 minutes. NPL will elute at 18 to 19 mins and EGDN will elute 1 to 2 mins after the NPL

Note: N.M.Liszt suggested the use of Fe^{++} for determination of purity of EGDN, but he did not describe the procedure

4.3.3 Moisture Content Determination by Karl Fisher (KF) Method.

Weigh accurately a 5 to 10g portion of the sample (sample size shall be in accordance with the strength of KF reagent) into a tared narrow-necked flask provided with a stopper. Titrate directly with standardized KF reagent until a brown tinge persists in the soln for 30 seconds

Note: Potentiometric end point indicators may also be used in titrations

Calculate as follows:

$$\% \text{ Moisture} = \frac{100\,(VF)}{W}$$

where: V= Volume in milliliters of KF reagent used in titration

F= Reagent Factor (grams of water per ml of reagent)

W= Weight of liquid proplnt in grams

Note: Detailed description of Karl Fisher Method is given in Vol 5, pp D1622 to D1628-L

4.3.4. Ignition or Explosion Temperature Determination. It shall be detd using differential scanning

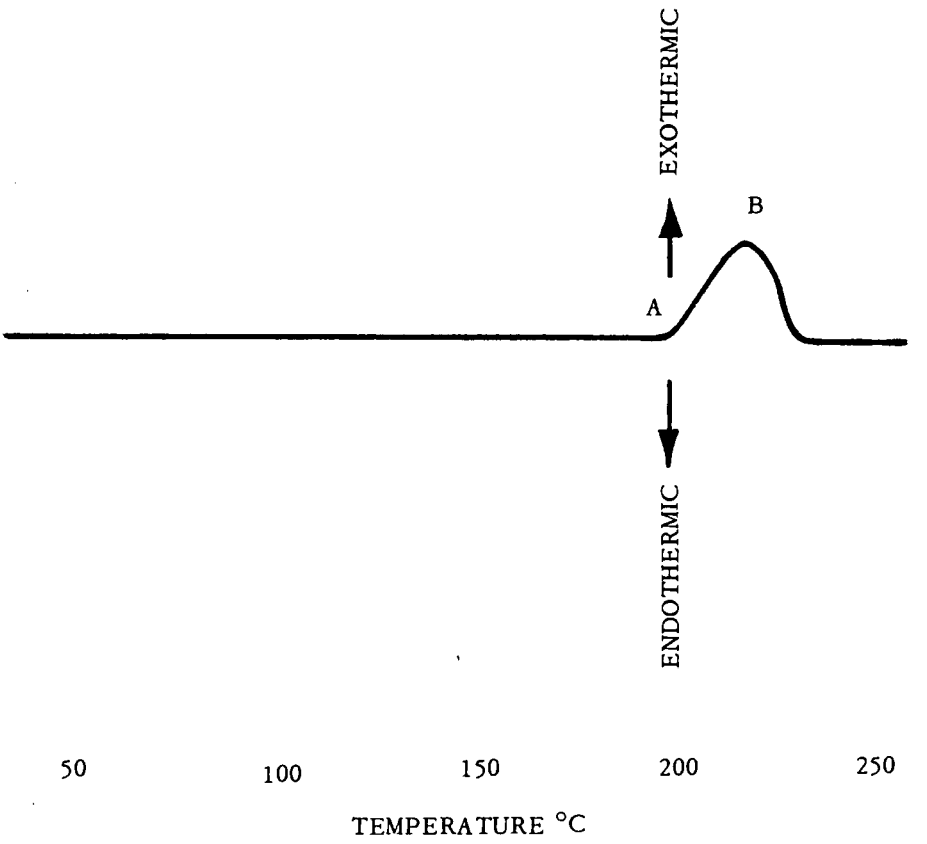


FIGURE Et19 INITIATION OR EXPLOSION TEMPERATURE DETERMINATION

SCAN SPEED 20°C/MIN
RANGE 8 CAL/SEC
CHART SPEED 40mm/MIN

calorimetry. The Perkin-Elmer Model DSC-1B Calorimeter with a number 219-0062 volatile sample sealer accessory may be used in conjunction with a Perkin-Elmer Number 196 Recorder

Procedure: Place ca 0.25mg of the liq proplnt in a volatile sample pan and seal the pan on volatile sample sealer accessory. Place sample and reference pans in their respective sample holders on the differential scanning calorimeter. Select program heating rate of 20°C per min, range 8 calories per sec, and recorder chart speed of 40ml per min. Start test at RT and program to 250°C. Ignition temp is interpreted to be the point at which the exothermic degradation begins, point A of Fig Et 19. Peak degradation temp is indicated by point B

Note: Description of "Calorimeter, Calorimetry & Calorimetric Determinations" is given in Vol 2 of Encycl, pp C10 to C12

4.3.5. Thermal Stability Determination

Procedure: Filter a portion of liq sample thru two thicknesses of "S and S" No 604 filter paper (or equivalent) and transfer (by means of a pipet) a 2ml portion of the filtered material to each of three test tubes, which are 5.5 inches long, 0.5 in internal diam and 0.62 in outside diam. (Care should be taken during the transfer not to leave droplets of the liq on the sides of the test tubes). Stopper each tube by means of a new, tightly fitting cork thru which passes a tightly fitting glass rod equipped with a Pt wire to hold a strip of std KI-starch indicator test paper, ca 1 in long and 3/8 in wide. Moisten a horizontal section in the upper half of the test paper with 50% (by vol) of pure glycerin in distd w. This can be done by dipping

a small diam glass rod into the glyc-w soln, and as the rod is withdrawn, contact is made with the side of the container so as to reduce the amt of soln adhering to the rod. Then draw the lower part of the rod across the paper strip so as to produce a level and distinct line of demarcation on the lower edge of the wet area. Prepare at the same time a blank by suspending a moistened strip of the same test paper in the clean dry test tube of the same size as tubes with samples. Adjust the temp of the heat tube bath to 82.2 ±1°C and insert all four tubes simultaneously into the heating soln to a depth of ca 2 inches. Note the time of insertion of the tubes and place the bath in such a position that the test tubes are viewed against a white background illuminated by bright diffused daylight. During the test, the line of demarcation of the papers in all tubes should be 3 inches above the level of liquids in test tubes. Consider the end point of the test to be the first appearance of any discoloration at the line of demarcation betn the wet and the dry portion of the paper in the sample tube which is in excess of the discoloration observed on the test paper of the blank. Note the time of completion of the test to the nearest minute. Consider the lowest time for the three tubes to represent the heat test value of the sample . As was mentioned above the time must be not lower than 8 minutes for NPL or EGDN

Note: After completion of the test empty the tubes and rinse them, as well as rods and corks, with acetone, followed by warm soapy tap w and distd w. Dry in a steam oven at ca 80°

4.3.6 Determination of Concentration of 2-Nitrodiphenylamine (2-NDPA) in EGDN and in Liquid Propellant

Weigh 2.00mg of Specification grade 2-NDPA to the nearest 0.02mg on a semimicro analytical balance and transfer to a 100ml volumetric flask. Add 4 drops of concd HNO_3 to dissolve the sample, dilute to volume with concd H_2SO_4 and mix well. Prepare standards by transferring 5.0, 2.0 and 1.0 microliter portions of this soln using a 10 microliter syringe to a 10ml volumetric flask. Dilute to volume with concd H_2SO_4 and mix well

Transfer 5.0 microliters of the sample (EGDN or liquid propellant), using a 10 microliter syringe to a 10 ml volumetric flask, dilute to volume with concd H_2SO_4 and mix

Measure the absorbance of the above standards and of the sample using 1.00 centimeter cells, simultaneously with absorbance of concd H_2SO_4 serving as a blank at 540 millicrons (nanometers). Use a suitable spectrophotometer, such as Beckman Model DU

Calculate the absorptivity from each of the standard solns, using the following equation and average the results

$$a = A/C$$

where:

a= Absorptivity; A= Absorbance (measured optical density) and C= concentration in micrograms per 10ml soln. In the above standards, 1 m microliter of stock soln contains 0.002 microgram of 2-NDPA

Calculate the concn of 2-NDPA in the sample using the following equiation

$$\%2\text{-NDPA} = 20\ A/a$$

where:

A= Absorbance = $\log_{10} 1/T$ (T is Transmittance - ratio of radiant power transmitted by sample to radiant power incident to it) and a = Mean Absorptivity of above compound

Remark: Mr. N.M.Liszt suggests the use of larger weights in detn of 2-NDPA and trying to find less reactive and dangerous solvents than concd HNO_3 and H_2SO_4. He does not know where the above procedure for 2-NDPA was developed, but both the concn and the wavelength differ greatly form standard method described in MIL-STD-286 for spectrophotometric determination

Note: Our rewrite of proposed specification was reviewed by Mr Nathan M. Liszt Chief, Analytical Branch, Propellants Div, FRL of Picatinny Arsenal

Ethyleneglycol Dinitrate–Nitroisobutylglycol Dinitrate Mixture. A soln of 10 parts NGc serving as an antifreeze in 90 parts of Nitroisobutylglycol Dinitrate (2-Methyl-2-nitro-1,3-propanediol Dinitrate) $CH_3C(NO_2)(CH_2ONO_2)_2$, was prepd and examined during WWII by the Hercules Powder Co, Wilmington, Delaware. It was found to be a powerful expl with the following properties: Density 1.48 at 28° and Detonation Velocity (when measured in a glass tube 1 cm ID and 1 mm wall thickness) 2050 m/sec & 7320 m/sec. It was suggested as a substitute for NG

Ref: G.D. Clift, formerly of Hercules Powder Co (1950)-private communication

Ethyleneglycol Dinitrate Polymers, $O_2NO.(CH_2CH_2OCH_2CH_2)_n ONO_2$. Two polymers were prepd by Oehme while nitrating

Ethylene oxide (qv) in presence of sulfuric acid: a) An oily substance contg 12% nitramine N and (b) A solid contg 9.3% of nitrometer nitrogen. Both were expls of low power. (See also Ethyleneglycol (Polymers) or Polyglycols)
Refs: 1) Beil 1, [521] 2)H.Oehme & Chem Fabrik Kalk GmbH, GerP376000 (1920) 3)Blatt OSRD 2014 (1944) 4)Curme & Johnston (1952)-not found

Ethyleneglycol Diphenylether
Ethyleneglycol-di-trinitrobutyrate
Ethyleneglycol Ethylether
Ethyleneglycolglycolate Dinitrate
Ethyleneglycol Lactate
Ethyleneglycol Lactate Dinitrate
Ethyleneglycol Methylether
Ethyleneglycol Methylether Acetate
Ethyleneglycol Monoalkyl Ethers
Ethyleneglycol Monoethers
Ethyleneglycol Monolactate
Ethyleneglycol Monolactate Acetate
Ethylene glycol Monomethyl Ether
Ethyleneglycol Mononitrate

See all of above items under ETHYLENEGLYCOL NITRITES AND NITRATES

Ethyleneglycol Monophenylether. See Ethyleneglycol Phenylether

Ethyleneglycolonitrile Nitrate. See Glycolonitrile Nitrate

Ethyleneglycol Phenylether and Derivatives
Ethyleneglycol Monophenylether, Phenyl "Cellosolve" or β-Phenoxyethanol; $C_6H_5O.CH_2CH_2.OH$; mw 138.17 col liq with faint aromatic odor; sp gr 1.094 at 20-20°, n_D1.534 at 20°, fr p 14° bp 240-248°, fl p 240°F, vapor pressure 0.03mm at 20°; sl sol in w, sol in alc & eth. Can be prepd by heating phenol & ethylene oxide in a closed tube at 150°. Used as a solvent for cellulose acetate, resins, dyes and inks. Its toxicity is reported in Ref 3, p 306 and in Ref 5. On its nitration were obtd the expls Ethyleneglycol Dinitrophenylether Nitrate (qv) and Ethyleneglycol Trinitrophenylether Nitrate (qv)

Note: Phenyl "Cellosolve" is Trademark of Union Carbide Corp
Refs: 1)Beil 6, 146, (84) & [150] 2) E.Roithner, Monats 15, 674, 678 (894) 3) Curme & Johnston (1952), 116 & 306 4) CondChemDict (1961), 469-L 5)Sax (1968), p 748

Ethyleneglycol-2,4-dinitrophenylether Nitrate, $(O_2NO)CH_2.CH_2O.C_6H_3(NO_2)_2$; mw 273.17, N15.39%, OB to CO_2–67.4%; crysts, mp 64-67°; heat of combstn at C_v 3455 cal/g, heat of formation 181 cal/g. Can be prepd by nitrating ethyleneglycol monophenylether. It is an expl sl less powerful than TNT and very insensitive to impact. It was proposed in Germany for use in smokeless proplnts & detonators
Refs: 1)Beil, not found 2)Westfälisch-Anhaltische Sprengstoff AG, ChemFabrik, Ger P548426 & CA 26, 3670 (1932) 3) Ibid, Ger P551306 & CA 26, 4474 (1932) 4) Schmid, SS 29, 263 (1934) 5) Curme & Johnston (1952) - not found

Ethyleneglycol-2,4,6-trinitrophenylether Nitrate, called Nitrade de 2,4,6-Trinitrophenylglycol in Ref 4) $(O_2NO)CH_2.CH_2.O.C_6H_2(NO_2)_3$; mw 318.16, N 17.61%, OB to CO_2, -45.3%. Crysts mp 103.3°(Ref 4); heat of combstn at C_v 291.8kcal/mol; heat of formation at Cv 66.7kcal/mol. It can be prepd by nitration of Ethyleneglycol Dinitrophenylether Nitrate or of Ethyleneglycol Monophenylether

It is an expl more powerful than TNT. It was proposed in Germany for use in detonators and in smokeless proplnts
Refs: 1) Beil, not found 2)Westfälisch-Anhaltische Sprengstoff AG, ChemFabrik, GerP548426 & CA 26, 3670 (1932) 3) Ibid, GerP 551306 & CA 26, 4474 (1932) 4)L. Médard & M.Thomas, MP 39,345-46 & 354 (1951)

Ethyleneglycol Polymers Nitrated. Propellants contg nitrated polyglycols are described in Ref. Tranchant prepd slabs of 60/40 CP_2 SD

(NC contg 11.6–11.7% N. See Vol 3, p C551-R)/ nitrate of polyglycols (triethylene-; tetraethylene-; pentaethylene- or hexaethylene-glycol). The polyglycol nitrates were emulsified in water before incorporation with the NC. A clear decrease in gelatinization power of the nitrates was observed with an increase in the chain length of the glycol
Ref: J. Tranchant, MP **32**, 287–89(1950)

Ethyleneglycol Urethanes. See under Glycol Urethanes

Ethyleneguanidine and Derivates
N,N'-Ethyleneguanidine, Imidazolidone-2-imide, *C(=NH)-NHCH$_2$CH$_2$NH; or Imidazoline-2-amine,* C(NH$_2$)=NCH$_2$CH$_2$NH; mw 85.11, N49.37%, The compound has only been prepd in manners which involve salt formation; attempts to isolate it resulted in formation of ammonia and ethyleneurea. First prepd (Ref 2) by heating at 100° in a closed tube ICN and ethylene diamine in alc. More recently ethylene diamine has been heated with a salt of 2-CH$_3$-2-thiopseudourea in water at 55° (Ref 5). Among the known acid salts are the **nitric**, prisms, mp 115° sol in w & alc, & the **picric**, mp 217°. Also known are a **mono-** and a **di-silver** salt, neither especially stable
Refs: 1)Beil **24**, 3&(184) 2)M. Schenck, ArchPharm **247**, 497(1910)& CA **4**, 1475(1910) 3)P. Pierron, AnnChim **11**, 361(1919) & CA **13**, 2022(1919) 4)D. Stefanye & W.C.Howard, JACS **77**, 761-2(1955) & CA **50**, 1780(1956) 5)L.S. Hafner & R.Evans, JOC **24**, 1157-9 (1959) & CA **54**, 5689(1960) 6)B.Adcock, et al, JCS **1961**, 5120-7 & CA **56**, 11577(1962) 7)H.Rapoport & J.V.Rodricks, JOC **1971**, 36(1), 46–8 & CA **74**, 42343(1971)
Note: The following derivatives of ethylene guanidine apparently are capable of being isolated in the isomeric forms indicated, plus A(i) mino-or nitra (i) minoethyl guanidines (non-cyclic) & not indicated, depending upon pH, thermal and temporal history of their method of prepn

2-Nitriminoethylenequanidine or 2-Nitriminoimidazolidine,
C(=N.NO$_2$)NH.CH$_2$.CH$_2$.NH; mw 130.11, N43.06%, mp 220-1° (dec), dip nom 6.93D, sol in water. Prepd by treating nitroguanidine with ethylenediamine dihydrochloride in w at 65-70° in the presence of KOH (Ref 2,4). More recently, ethylene diamine was reacted with 2-methyl-1-nitro-2-thiopseudourea (also a prepn for ethyleneguanidine without the nitro)(Ref 8). Treatment with sulfuric or perchloric acids converts this compound to the **nitric acid salt of 2-aminoimidazole**, mp 112-13° (Ref 7)
Refs: 1)Beil, not found 2)A.F.McKay & G.F.Wright, JACS **70**, 430-1 (1948) & CA **42**, 2253(1948) 3)A.F.McKay, et al, JACS **71**, 766-70 (1949)& CA **43**, 4273 (1949) 4) A.F.McKay & G.F.Wright, USP 2525927(1950) & CA **45**, 2512(1951) 5)W.D.Kumler, JOC **18**, 676-9(1953) & CA **48**, 6969(1954)(Dipole moment study) 6)W.D.Kumler, JACS **76**, 814-16(1954) & CA **48**, 8051(1954) Infrared spectral data) 7)T.G.Bonner & J.C.Lockhart, JCS **1958**, 3852-8 & CA **53**, 15063 (1959) 8)L.S.Hafner & R. Evans, JOC **24**, 1157-9(1959) & CA **54**, 5869(1960)

2-Nitraminoimidazoline-2,
C(N.HNO$_2$)=N.CH$_2$.CH$_2$.NH; mw 130.11, N 43.06%. Barton (Ref 2) showed that this isomer is formed when an alkaline solution of the preceding entry is allowed to stand a day or so. Apparently there is no literature mention of its actual isolation, although the earlier Refs for the preceding entry use the (a)amino structure
Refs: 1)Beil, not found 2)S.S.Barton, et al, JACS **73**, 2201-5(1951)& CA **46**, 1988 (1952)

1-Nitroethyleneguanidine or 1-Nitro-2-Iminoimidazole,
C(= NH)NHCH$_2$CH$_2$N(NO$_2$); mw 130.11, N43.06%, mp 191–2° (may be the mp of an HCl salt, given in Ref 3), sol in water. Other than, possibly, Ref 3, this compound seems to have been prepd only as acid salts. McKay (Ref 2) prepd the **HCl salt**, mp 187.5-8.0° (dec), by reacting chloroethylamine with 1-methyl-1-nitrosonitroguanidine to give chloroethylnitroguani-

dine which was heated in water to close the ring. In the same paper McKay reports the **HNO_3 salt**, mp 161° (dec) prepd by treating the HCl salt with $AgNO_3$, by heating with $AgNO_3$ the acyclic intermediate to the HCl salt, or by nitrating hydroxyethylnitroguanidine to nitroethylnitroguanidine and heating to close the ring. Most recently cyanogenbromide was reacted with

$CH_2CH_2NC \leq {NH_2 \atop N.NO_2}$

to give the **HBr salt** (Ref 4)

Refs: 1)Beil, not found 2) A.F.McKay & J.E. Milks, JACS **72**, 1616-20(1950) & CA **44**, 10661(1950) 3)W.A.Skinner et al, JMedPharm Chem **2**, 299-333(1960) & CA **54**, 22595(1960) 4)J.A.Lowe, et al, JOC **28**, 1496-8(1963) & CA **59**, 1619(1963)

1-Nitro-2-Aminoimidazoline-2,
$C(NH_2)=NCH_2CH_2N(NO_2)$; mw 130.11, N43.06%. See preceding entry and its Refs and note at beginning of this series of compounds

1-Nitro-2-Nitriminoethyleneguanidine or 1-Nitro-2-Nitriminoimidazolidine,
$C(=NNO_2)NHCH_2CH_2NNO_2$; mw 175.10, N 40.00%, OB to CO_2 −41.1%, mp 151-2° (dec). Prepd by nitration in sulfuric or acetic/acetic anhydride of the 2-nitriminoethyleneguanidine compd (see above)(Ref 2). Kumler (Ref4) states that the dipole mon of 7.65D and the pKa of 6.3 indicate this to be the structure "normally" rather than that in the following entry. Treatment with ammonia followed by HCl open up the ring giving chloroethylnitroguanidine (Ref 6). Treatment with $AgNO_3$ in aq alc give a **silver salt**, which expl over an open flame (Ref 7)

Refs: 1)Beil, not found 2)A.F.McKay & G.F.Wright, JACS **70**, 3990-4(1948) & CA **43**, 2203(1949) 3) A.F.Mckay & W.G.Hatton, ibid **75**, 963-5(1953) & CA **48**, 2049(1954) 4)W.D.Kumler, JOC **18**, 676-9(1953) & CA **48**, 6969(1954) 5)W.D.Kumler, JACS **76**, 814-16 (1954) & CA **48**, 8051(1954)(Infrared spectral data) 6)A.F.McKay, ibid, **77**, 1057-8(1955) & CA **49**, 8929(1955) 7)A.F.McKay et al, ibid **76**, 6371-4(1954) & CA **49**, 15861(1955)

1-Nitro-2-Nitraminoimidazoline-2,
$C(NHNO_2)=NCH_2CH_2N.NO_2$; mw 175.10, N40.00%. See preceding entry and its Refs and Note at beginning of this series of compounds

1-Nitro-2-Amino-2-Nitraminoimidazole,
$C(NH_2)(NHNO_2)NHCH_2CH_2N.NO_2$; mw 192.135, N43.74%, OB to CO_2-50.0%, crysts (from water) mp 184.8 - 5.3° (dec). Prepd by adding amonia to the 1-nitro-2-nitrimino compd in a non-aq system; if water is present, the compd does not form or converts to nitraminoethylnitroguanidine

Refs: 1)Beil, not found 2)A.F. McKay & G.F.Wright, JACS **70**, 3990-4(1948) & CA **43**, 2203 (1949) 3)R.S.Hall et al, ibid, **73**, 2205-8(1951) & CA **46**, 1988(1952) 4)M.W.Kirkwood & G.F.Wright, JOC **18**, 629-42(1953) & CA **48**, 6968 (1954)

1-Nitroso-2-Nitriminoethyleneguanidine or 1-Nitroso-2-Nitriminoimidazolidine,
$C(=NNO_2)NH.CH_2CH_2N.NO$; mw 159.10, N44.02%, OB to CO_2-55.3%, mp 141-2°. Prepd by treating the 2-nitrimino compd with nitrous acid (Ref 2). Treatment with aqueous methylamine at 5-7° displaces the =NO with an −H (Ref 4)

Refs: 1)Beil, not found 2)A.F.McKay et al, JACS **72**, 3659-61(1950) & CA **45**, 2888 (1951) 3)A.F.McKay, JOC **16**, 1395-1404 (1951) & CA **46**, 5584 (1952) 4)A.F.McKay, CanJChem **31**, 284-6(1953) & CA **48**, 153 (1954) 5) W.A. Skinner et al, JMedPharm-Chem **2**, 299-333(1960) & CA **54**, 22595(1960)

1-Nitroso-2-Nitraminoimidazoline-2
$C(NHNO_2)=NCH_2CH_2N.NO$, mw 159.10, N44.02%. See preceding entry and its Refs and note at beginning of this series of compounds

Ethyleneimine and Derivatives

Ethyleneimine or Aziridine, CH_2CH_2NH; mw 43.07, N 32.52%, frp-74°, Bp 55-6°, sp gr 0.8321 at 24°, n_D 1.4123 at 25°, sol in w, & organic solvs. Prepd by treating 2-bromoethylamine hydrobromide with silver oxide (Ref 2). Originally thought to be vinylamine, and not correctly identified until 11 years later (Ref 3). In 1963 Dow Chemical introduced the method

of prepn by heating ethylene dichloride with ammonia(in excess or with CaO) at 100°. Most salts are unstable at room temperature, giving ring opening, but the **picrate** is stable, mp 142°,yel. Ethyleneimine is extremely toxic and corrosive to all tissue (Ref 6)
Refs: 1)Beil **20**, 1,(3) & [3] 2)S.Gabriel, Ber **21**, 1049(1888) 3)C.C.Howard & & W. Marckwald, Ber **32**,2036(1899) 4) A.Weissberger, ed,"Chemistry of Heterocyclic Compounds", Interscience-Wiley, NY **19**,(1964), p 524-L(P.Fanta) 5)"Ethylenimine", The Dow Chemical Co, Midland, Mich (1965) 6)K&O, **11**,(1966),pp 526-548 (O.C. Dermer & A.W. Hart, 163 refs) 7) Sax(1968),748

N-Nitrosoethyleneimine or N-Nitrosoaziridine, CH_2CH_2NNO; mw 72.07, N 38.86%. Prepd by adding NOCl dropwise to ethyleneimine, add triethylamine in eth at -70°. The resulting yel soln is stable at -60°, but decolorizes on warming. Above 0° decompn to ethylene and nitrous oxide occurs
Refs: 1)Beil, not found 2)W.Rundel & E. Mueller, Ber **96**,(9),2528-31(1963) & CA **59**, 12626(1963)

Note: No other derivatives of ethyleneimine of the type NO_x or N_3 were found in Beil or through CA **75**

Ethylene Nitrate See under Ethylene Glycol Dinitrate in this volume

Ethylene Nitrite See under Ethylene Glycol Dinitrite in this volume

Ethylene Nitro, $O_2NCH{=}CHNO_2$; mw 118.05, N23.73%, OB to CO_2-13.5%, mp 64°. Prepd by treating an ether suspension of the disodium salt of 1,2-dinitroethane at -50° with bromine It is not stated in any of the refs whether it is the cis or trans isomer
Refs: 1)Beil, not found 2)E.S.Lipina, et al, ZhOrgKhim **5**(7), 1312–13(1969) & CA **71**, 101212(1969) 3) Ibid, RussP 253039(1969) & CA **72**, 120999(1970) 4) Ibid, DoklAkad-Nauk(Russ) **192**(4), 810–12[Chem](1970) & CA **73**, 55570(1970)

Ethylene Nitrosite. See Ethylene Nitrosite in this Vol

Ethyleneoxamide and Dinitroethyleneoxamide. They are described in Vol 5 of Encycl,pp D1299-R & D1300-L, under Diketopiperazine and Derivatives

A different method for prepg ethyleneoxamide than that given on pD1299-R was described by J. van Alphen, Rec **54**, 937(1935), who also described on p 938 prepn of N,N'-Dinitroethyleneoxamide listed on p D1299-R of Encycl as 1,4-Dinitrodiketopiperazine

Another method of prepn of Dinitroethylene oxamide was described by W.E.Bachmann, JACS **72**, 3133(1950). He prepd it by stirring gradually 15g of ethyleneoxamide into a freshly prepd mixture of 40g 98%, white nitric acid and 40g acetic anhydride, kept at 5 °C or below. Then the nitrated mixt was placed in a refrigerator and kept there for 20 hours. The resulting ppt was collected on a sintered glass filter and rinsed with acetic acid. After drying, 16.6g of product melting at 180-185° was collected
Bachmann also obtd 5.2g of EDNA by warming for a few minutes on a water bath, 8g of Dinitroethyleneoxamide in 10 ml of 28% of ammonia

ETHYLENE OXIDE (EtnO) Epoxyethane or Dimethylene Oxide, $H_2C{-}CH_2$ (O); mw 46.06; pleasant smelling, flammable liquid, sp gr 0.8697 at 20°; fr p -111.3°, bp 13.5° at 747mm; N_D1.364 at 6.4°or 1.3597 at 7°, flash p(tag glass open cup) <0°F; coeff of expansion 0.0017 at 55°, dielectric constant 13.9 esu at -1°C; ignition temp in air at 760mm 429°C (Perkin); spontaneous ignition temp (SIT) 804°F (429°C);[IEC **32**,882(1940)]; vapor pressure at 20° 1095mm; viscosity (absol) at 0° 0.32cp; surface tension 24.3 dynes at 20°; heat of combstn at 25° & 760mm 312.55 ± 0.20 kcal/mol; heat of formation-vapor 17kcal/mol & liquid 23.3kcal/mol; heat of fusion 1236.4cal/mol; heat of vaporization at

760mm and 10.5°C 6082cal/mol- thermal conductivity of vapor at 25° 0.00002961cal-cm/sec-/cm^2/°C. It does not corrode metals

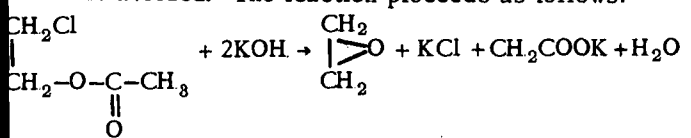
EtnO is very sol in w, alc, ether, dichloroethane and other org solvents. Although aqueous solns of EtnO conduct electricity, the compd itself is not conductive; when, however, a salt like NaCl or KNO_3 is dissolved in EtnO, the resulting soln is conductive

Physical properties of EtnO are listed on pp 87-94 of Curme & Johnston's book (Ref 17), while its chemical reactions are described on pp 94-106

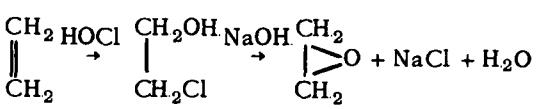
Wurtz (Refs 1 & 2) was the first to prep it in 1859, thus laying the foundation for the "chlorohydrin process" currently used in industry. A procedure by which oxygen can be added directly to the ethylene bond was discovered in 1931 by Lefort and patented in 1935 (Ref 6a). His research led to the development of a second major process for EtnO manuf, known now as "direct oxidation" (Ref 17, p 74)

The Carbide and Carbon Co began large scale manuf of EtnO thru ethylene chlorohydrin in 1925 and by the direct oxidation of ethylene in 1937. Dow entered the field in 1939 & 1941; Jefferson and Wyandotte in 1941 and Mathieson Chem Corp in 1951. These four Co's used the chlorohydrin method. US consumption, which was in 1939 108 million pounds, increased in 1949 to 354 million

Several laboratory methods of prepn are described in Ref 17, pp 75–7. In one of them hydroxide is added gradually to a soln of 2-chloroethyl acetate heated to a temp betwn 40 & 150°C. An excess of unreacted base is avoided. The reaction proceeds as follows:



Commercial prepn of EtnO by chlorohydrin method is based on the following reaction:



This method is described in Ref 17, pp 16–19 & 77 and in Ref 13

CaOH can be used instead of NaOH, as described by Messing (Ref 13a)

The direct catalytic, oxidation method of ethylene is described in Ref 17, pp 77–87: *Explosibility.* Liquid ethylene oxide is stable to detonating agents, but the vapor will undergo explosive decomposition. Pure ethylene oxide vapor will decompose partially; however, a slight dilution with air or a small increase in initial pressure provides an ideal condition for complete decomposition. Copper or other acetylide-forming metals such as silver, magnesium, and alloys of such metals should not be used to handle or store ethylene oxide because of the danger of the possible presence of acetylene. Acetylides detonate readily and will initiate explosive decomposition of ethylene oxide vapor. In the presence of certain catalysts, liquid ethylene oxide forms a poly-condensate. This reaction is highly exothermic. If the heat of the reaction is not conducted thru the walls of a closed container at a rate capable of maintaining an equilibrium temperature, an increase in pressure results with an increase in reaction rate, leading to explosive conditions. Acid salts, such as stannic chloride and zinc chloride, and bases, such as alkali metal hydroxides, either solid or in aqueous solution, and tertiary amines are all effective catalysts. It is, therefore, imperative that the concentration of such contaminants be kept at a minimum when transporting or storing sizeable quantities of ethylene oxide

Accdg to Hess & Tilton (Ref 16), a 90% decompn takes place if 100% vapor of EtnO in a closed container is initiated with MF. There is no upper limit of EtnO in air (the previously reported value of 80% was in error), but the lower expl limit is 3% (Ref 17, p 87)

Toxicity of EtnO (Ref 17, pp 314–15 & Spec MIL-E-52171). Liquid EtnO, concentrated or dilute, when exposed to the skin can cause severe delayed burns. Short exposures produce mild first degree burns, but prolonged exposures produce second degree burns with the formation of large blisters. Exposure to the vapor results in systemic manifestations and irritation to the respiratory system. Inhalation of ethylene oxide vapors, if prolonged, results in severe systemic poisoning with the symptoms of nausea, vomiting, headache, dysnea, and diarrhea. The anesthetic properties are similar to chloroform, but with pronounced undesirable side and after effects.

Tests on guinea pigs have shown that a 5 percent by volume concentration of ethylene oxide vapor kills in a short time. The maximum concentration for 60 minutes without serious disturbance was 0.3 percent by volume. The odor of EtnO is inadequate to serve as a warning of dangerous concns. Some authorities consider that concns as low as 250ppm have some effects on humans and that 500ppm is dangerous. Concns used for fumigation are not highly toxic, but they can cause cyanosis. Skin exposure to liquid EtnO can cause severe delayed burns

Uses of EtnO: It has been used chiefly as an intermediate for prepn of other compds, such as of ethyleneglycol, other glycols, alcohols, etc (Refs 13, 17 & 18). Its hydration to ethyleneglycol is described in Ref 17, p 20

EtnO has also been used as a disinfectant or fumigant (Ref 17, p 108 & Ref 18). Technical EtnO mixed with some carbon dioxide or chlorofluoro hydrocarbons (to remove the expln hazard) is intended for use as a biological decontaminant (See Ref 19). Nearly pure (99.9%) EtnO is used as a liquid propellant (Ref 21). Its use as a monopropellant is discussed in AddnlRefs I & J

Analysis of EtnO and its homologues:

Accdg to Curme & Johnston (Ref 17, p97), with certain metallic halides in neutral aqueous soln, EtnO reacts with the acid freed by dissociation and hydrolysis to ppt the metallic hydroxide. This reaction serves as a basis of analysis for EtnO and its homologues. It proceeds as follows:

$$MgCl_2+2H_2C\!-\!CH_2+H_2O \rightarrow 2ClH_2C.CH_2OH+Mg(OH)_2$$

The halides of aluminum, iron, tin, zinc and manganese react in similar manner with EtnO in neutral aqueous soln

Deckert (Ref 6, quoted from Ref 17) developed a qualitative test for EtnO based on the reaction with aqueous NaCl which results in the formation of ethylene chlorohydrin and NaOH

Lubatti (Ref 8, quoted from Ref 17) developed a quantitative test for EtnO using HCl in a saturated soln of $MgCl_2$

The nitration of EtnO in the presence of sulfuric acid yields Ethyleneglycol Dinitrate:

$$\begin{matrix} H_2C \\ | \\ H_2C \end{matrix}\!\!>\!O+2HNO_3 \xrightarrow{H_2SO_4} \begin{matrix} CH_2ONO_2 \\ | \\ CH_2ONO_2 \end{matrix} +H_2O$$

(Ref 17, p 104 & AddnlRef F)

Refs for EtnO:

1) Beil **17**, 4, (3) & [9] 2) A. Wurzt (pronounced Würtz), AnnChim et Phys (3), **55**, 433(1859); Annalen **110**, 125(1859) and Bull(Fr) (2), **29**, 530(1878) 3) W.H. Perkin, JCS **63**, 488(1893) 4) O. Mass & E.H. Boomer, JACS **44**, 1709(1922) 4a) Naoúm, NG(1928), 222 5) A.J. Headle, "A Study of Ethylene Oxide and Some of Its Derivatives", Thesis, West Virginia Univ (1932) 6) W. Deckert, AngewChem **45**, 559 (1932)(Qualitative determination of ethylene oxide) 6a) T.E. Lefort (to Société Française de Catalyze Généralisé), USP 1998878 (1935) 7) C. Ellis, "The Chemistry of Petroleum Derivatives", Reinhold, NY, **1** (1934) & **2**(1937) 8) O.F. Lubatti, JSCI **54**, 424T(1935)(Quantitative determination of ethylene oxide) 9) R.S. Crog & H. Hunt, JPhysChem **46**, 1162(1940) 10) J.G. Kern, R.L. Murray & R.W. Subhoff, PB Rept No 485(1945) 11) R.M. Goepp, FIAT Final Rept **N874**(1947) 12) W.F. Giauque & J Gordon, JACS **71**, 2176–2181(1949) 13) J.R. Skeen, ChemEngrg **57**(7), 331(1950) 13a) R.F. Messing, ChemInds **67**, 41(1950) 14) R.E. Kirk & D.F. Othmer, "Encyclopedia of Chemical Technology", Interscience, NY, **5**(1950), p 922 15) W.L. Faith, D.B. Keyes & D.L. Clark, "Industrial Chemicals", J. Wiley, NY(1950), pp 327–328 (under Ethyleneglycol) 16) L.G. Hess & V.V. Tilton, IEC **42**, 1251–58(1950)(Hazards and methods of handling ethylene oxide)(20 refs) 17) G.O. Curme, Jr & F. Johnston, Edit, "Glycols", Reinhold, NY(1952), pp 74–113: H.C. Schultze, "Ethylene Oxide" (175 refs) 18) CondChemDict (1961), 469-R 19) Military Sepcification MIL-E-52171, 27 Apr 1961 and Amendment 1, 6 Oct 1961 (Ethylene Oxide, Technical) 20) Kirk & Othmer **8**(1965), pp 523–558(H.C. Schultze) 21) Military Specification MIL-P-8845A, 30 June 1969 (Propellant, Ethylene Oxide) 22) Sax, 3rd edit (1968), 749 (Props, toxicity & hazards)

AddnlRefs for EtnO:

A) H. Moureu & M. Dode, BullSocChim [5], **4**, 637–47(1937) & CA **31**, 4884(1937) (Heat of formation) B) R.S. Crog & H. Hunt, JPhysChem **46**, 1162–63(1942) & CA **37**, 1324(1943) (Heat of combustion)

C) Koch, Arbeitschutz **1943**, 317 & CA **40**, 6259(1946) (Serious expln caused by EtnO is discussed) D) A.K. Gupta, JSCI **68**, 179–83(1949) & CA **43**, 8138(1949) (EtnO in the presence of small amts of acids, bases, or some salts might undergo a spontaneous polycondensation of explosive violence) E) Anon, Chem&EngrgNews **27**(35), 2500(1949)(Brief deecription of explns reported by Gupta and recommendations for prevention of such explns) F) Tanabe Chem Ind Co, JapanP 157836 (1943) & CA **44**, 3010(1950)(Nitration of EtnO) G) Bibliography of Reports on Ethylene Oxide, IB226, Office of Technical Services, OTS(1951) H) E. Wilson, JAmRocketSoc **23**, 368–72(1953) & CA **48**, 6126(1954)(The stability of EtnO) I) W. Robinson, JetPropulsion **24**, 111–12(1954) & CA **49**, 2074(1955)(EtnO as a monopropellant) J) J. Glasstone & J. Scott, JetPropulsion **24**, 386(1954) & CA **49**, 2075(1955)(EtnO as a monopropellant) K) P. Dittmar & A. Cantzler, Explosivstoffe **1957**, 259(Expln of EtnO) L) Anon, IEC **50**, 15 (Dec 1958) [Discussion of reasons for the expln of EtnO at Geismar, La plant of Wyandotte Chemicals, which was reported in C&EN, Dec 8(1958), p 25]

Ethylene Oxide, Technical

Military Specification **MIL-E-52171**, 27 April 1961 and Amendment 1, 6 October 1961. Material described in this test is used with added carbon dioxide or chlorofluorohydrocarbons (to prevent expln hazard) as a biological decontaminant

Requirements:	% by Weight
1) Assay [$(CH_2)_2O$]	99.0 min
2) Water	0.030 max
3) Acidity (as acetic acid)	0.002 max
4) Aldehydes (as acetaldehyde)	0.010 max
5) Chlorides (inorganic)	none
6) Acetylene	none
7) Nonvolatile residue	0.010g max (per 100ml)
8) Color	15 max on the Pt-Co scale

Tests: Tests shall be conducted as follows: Exercise care in the handling of ethylene oxide as it is an explosive and toxic material. Conduct all tests in a ventilated hood. Use analytical grade reagents and distilled water thruout the tests. Run blank determinations in parallel with the tests, using the same quantities of reagents, and apply corrections when significant. Determine the specific gravity of the specimen at 1°±1°C by means of a suitable hydrometer, and use the figure obtained for calculations

Preparation of specimen. Suspend a sample bottle and metal carrier similar to Fig Et 20 in a dry ice-acetone bath contained in a Dewar flask. Fill the bottle to the shoulder with a portion of the sample ethylene oxide and cover the bottle with a glass plate. Dry a borosilicate glass bulb 18 to 22mm in diameter having a narrow stem approx 50mm long in an oven at 105°±5°C, cool it in a desiccator, and weigh it to the nearest 0.1mg

Place the bulb in the glass dipper, and replace the glass plate with the bulb and dipper as shown on Fig Et 20. Cover the bulb in the glass dipper with carbon dioxide snow, and allow sufficient time to elapse for 1 g of the sample to be drawn into the bulb. Immediately remove the glass dipper by the handle, invert the bulb, and then hold the bulb stem at an angle of approximately 45° from the vertical. Bring the stem near a gas flame to warm slightly. When the liquid leaves the capillary, seal the bulb quickly with the gas flame while the bulb is still in the carbon dioxide snow

Procedure. Remove the bulb from the glass dipper. Allow the bulb to come to room temperature in a desiccator and weigh to the nearest 0.1mg. Place 150g of magnesium chloride, 125ml of saturated magnesium chloride solution, and 30.0ml of 1N sulfuric acid in a 500-ml heavy-wall, glass-stoppered Erlenmeyer flask. (Thirty milliequivalents of sulfuric acid are approximately equal to 1.34g of 99.0% ethylene oxide. If the normality of the sulfuric acid is much less than 1N, an additional quantity of acid should be added to make a total of 30 milliequivalents. Also, if the sample is 1.34g or more, additional acid and magnesium chloride should be added.) Introduce the sealed sample of ethylene oxide into the Erlenmeyer flask and stopper tightly, taking care not to break the bulb. (Since the flask must be sealed, the stopper should be reground with No 300 carborundum and greased.) Cool in an ice bath to 1°±1°C. Remove the

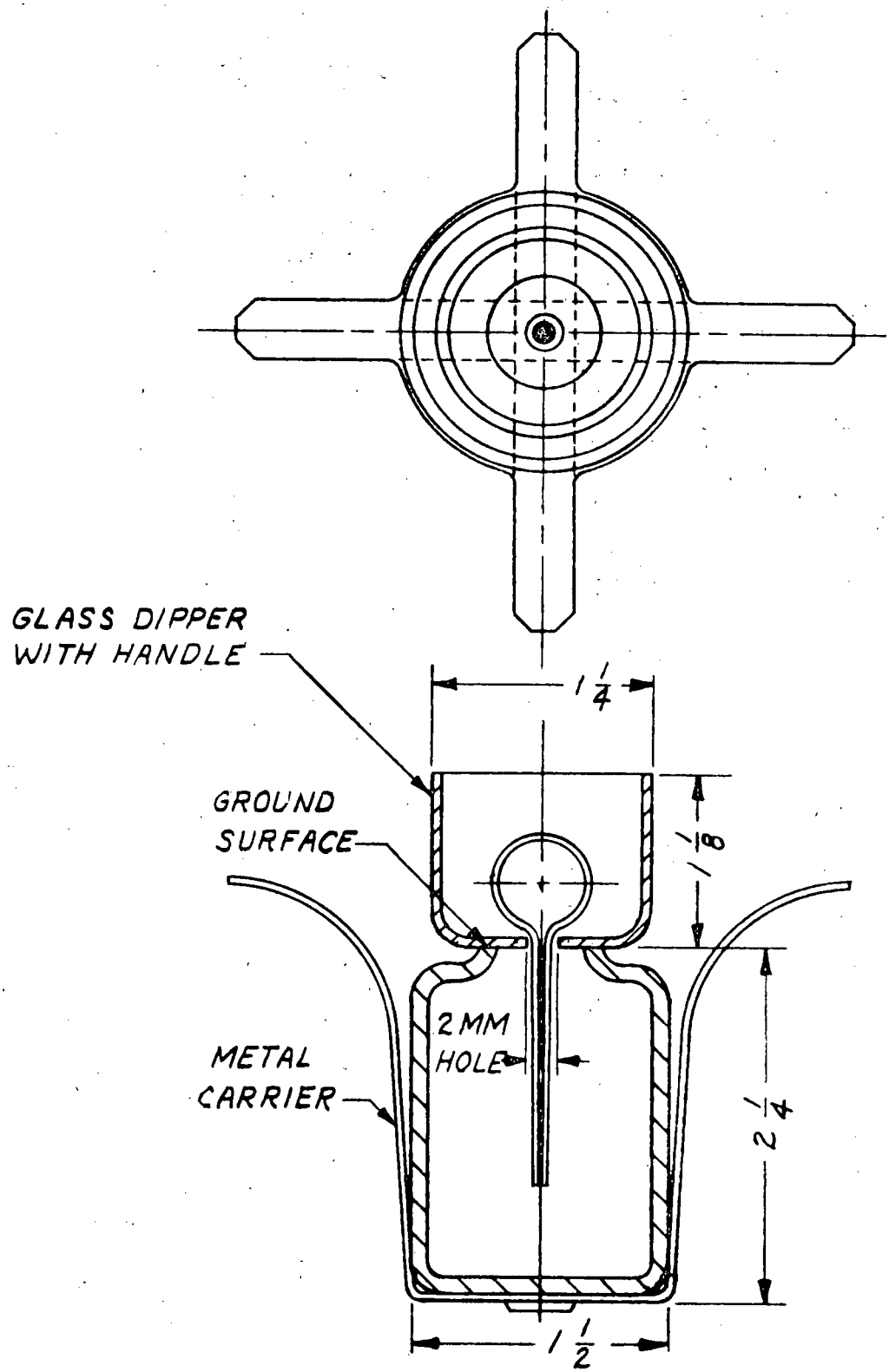


FIGURE E-20 BOTTLE AND METAL CARRIER

flask, wrap with a towel, and holding the stopper down tightly, shake to break the bulb. Allow the contents to react, shaking intermittently for 15 minutes. If the bulb stem is not completely shattered by shaking, crush the stem with a stirring rod. Titrate the excess acid with 0.1N sodium hydroxide to a bromcresol purple endpoint (yellow to blue). Run a blank titration on the reagents, using exactly the same amount of sulfuric acid. Calculate as follows:

$$\% \text{ EtnO} = \frac{(B-A)\ N \times 4.405}{g \text{ of sample}}$$

where: A = ml of NaOH soln required for the sample.
B = ml NaOH soln required for the blank
N = normality of the NaOH soln

Retest. Retest the lot if the results of tests of the composite and the individual samples vary over a range greater than 0.3%

Water. Determine the water content of a 50-ml specimen of the EtnO as specified in ASTM Standard D1364. Calculate the weight of the specimen tested using the specific gravity of the ethylene oxide determined by means of a suitable hydrometer

Acidity (as acetic acid). Using a chilled 100-ml graduate, measure a 68-ml (60g) portion of the sample into a 250-ml Erlenmeyer flask. Allow the portion to evaporate to about 50ml to release any carbon dioxide in the sample. Immediately add 3 drops of a 1.0% soln of phenolphthalein in a 1:1 mixture of methanol and water, and titrate with an aqueous 0.01N NaOH to a pink end point which persists for 15 secs. Calculate the acidity as acetic acid using the formula:

$$\% \text{ Acidity (as acetic acid)} = \frac{A \times N \times 6.0}{B \times G}$$

where: A = ml of 0.01N NaOH
N = normality of NaOH
B = ml of EtnO
G = specific gravity of sample

Aldehydes (as acetaldehyde). Reagents: Sodium metabisulfite solution (0.1N). Dissolve 4.75 to 5.00g of sodium metabisulfite in sufficient water to make 1000ml of solution. Adjust the pH of the solution to 3.5 by adding 0.5N sulfuric acid. Make up a fresh solution daily. Do not use this solution after the pH drops below 3.0

Iodine solution(0.1N). Dissolve 13.5g of iodine in 200ml of water containing 24g of KI. Dilute the solution to 1000ml with water. Standardize the soln against standard sodium thiosulfate or standard sodium arsenite solns

Procedure. Transfer 25.0ml of distilled water to each of four 500-ml glass-stoppered Erlenmeyer flasks and cool them to approx 0°C. Carefully pipet 20.0ml of the 0.1N sodium metabisulfate soln into each flask, using the same pipet for each transfer and holding the tip of the pipet under the surface of the water in the flask during delivery. Cool the contents of the flasks to approx 0°C, and set aside two of the flasks for blank determinations. Using a 50-ml graduate cooled by rinsing with portions of the cooled sample, pour 50ml of the sample into each of the other two flasks. Swirl the contents of the flasks to effect soln. Allow the four flasks to stand, at room temp, for at least 10 mins but not more than 30 mins with occasional swirling. Add 10ml of water previously cooled in an ice bath to each flask. Immediately titrate the contents of each of the flasks with 0.1N iodine solution until the yellow color begins to persist, add 5ml of freshly prepared 1.0% aqueous starch indicator, and continue the titration to the first permanent blue end point. Calculate as follows:

$$\% \text{ Acetaldehyde} = \frac{(B-A)\ N \times 2.2}{50 \times G}$$

where: A = ml 0.1N iodine soln required for sample
B = average ml 0.1N iodine soln required for blank
N = normality of iodine soln
G = specific gravity of sample

Chlorides (inorganic). Place 40ml of water in a 250-ml glass-stoppered Erlenmeyer flask and cool the water in an ice bath. Add approx 10ml of chilled EtnO sample, 10 drops of concd nitric acid, and about 10ml of approx 0.1N silver nitrate soln. Report the presence of inorganic chlorides if a ppt is formed

Acetylene. Reagents: Mixed indicator. Dissolve 0.10g of methyl red and 0.050g of methylene blue in 100ml of 95% ethyl alcohol conforming to grade I, class B, of Specification O-E-760. Mix the solution thoroughly and store it in a dark-colored bottle

Silver nitrate solution. Dissolve 25g of silver nitrate in 150ml of water contained in

a 1000-ml volumetric flask. Mix the soln thoroughly and dilute to the mark with 95% ethyl alcohol conforming to grade I, class B, of Specification O-E-760. Mix the soln thoroughly

Procedure. Transfer 150.0ml of the silver nitrate soln to each of two 250-ml glass-stoppered Erlenmeyer flasks. Place the flasks in a suitable ice bath until the temperature of the contents is 2°±2°C. Add 7 to 9 drops of the mixed indicator to each of the flasks and, if necessary, neutralize the soln to a gray-green color with 0.02N NaOH, if acid, or with 0.02N nitric acid if alkaline. Remove one of the flasks from the ice bath for the blank. Using a chilled graduate, pour 50±1 ml of the sample into the flask remaining in the ice bath. Swirl the flasks and compare the colors of the contents. Report the presence of acetylene if the soln containing the sample turns to a purple color. (If a positive test is obtd, destroy the silver acetylide by adding an excess of acid ferrous sulfate soln to the contents of the flask before discarding the soln)

Non-volatile residue. Measure approx 100ml of the cold sample into a chilled 100-ml graduate and place it in a tared borosilicate glass evaporating dish. Allow the EtnO to evaporate to dryness at RT. Place the dish in an oven at 105° to 110°C for 30 mins. Cool the dish in a desiccator and reweigh it to the nearest mg. Calculate the non-volatile residue by subtracting the tare wt from the wt of the dish containing the residue

Color. Standards: Stock standard. Dissolve 1.245g of potassium chloroplatinate (K_2PtCl_6) and 1.000g of cobaltous chloride ($CoCl_26H_2O$) in water. Add 100ml of concd HCl, and dilute the soln to 1000.0ml with water. (This soln has a color of 500 on the APHA and ASTM platinum-cobalt scales)

Test standard. Dilute 3.0ml of the stock standard to 100.0ml with water. (This soln represents a color of 15 on the Pt-Co scale)

Procecure. Fill one of a pair of matched tall-form 100-ml comparison tubes to the mark with the test standard. Chill the second matched tube and fill it to the mark with the chilled sample. Immediately compare the colors of the test standard and the sample by viewing vertically down thru the tubes using a white background. Report the sample as passing the requirement if the sample is no darker than the test standard

Acceptance/rejection criteria. If either sample fails to meet the requirements of this specification, the lot represented shall be rejected

Propellant, Ethylene Oxide

Military Specification MIL-P-8845A, 30 June 1969, superseding MIL-P-8845(ASG), 20 April 1959

Requirements:	% by Weight
1) Assay [$(CH_2)_2O$]	99.9 min
2) Acidity (as acetic acid)	0.005 max
3) Water	0.03 max
4) Iron, ppm/wt	0.1 max
5) Aldehyde (as acetaldehyde)	0.03 max
6) Chloride	0.02 max
7) Non-volatile residue	0.01 max
8) Acetylene	non detectable

Reagents Used in Tests:

A) *Mixed indicator.* Dissolve 0.10g of methyl red and 0.050g of methylene blue in 100ml of 95% ethanol. Mix and store in a brown bottle

B) *Silver nitrate solution.* Dissolve 25g of ACS reagent grade silver nitrate in 150ml distilled water contained in a 1000-ml volumetric flask. Mix thoroughly and dilute to the mark with 95% ethanol

C) *Sodium hydroxide solution, 0.02N.* Dissolve 0.8g of ACS reagent grade NaOH in distilled water in a 1000-ml volumetric flask. Dilute to the mark with distilled water

D) *Nitric acid solution, 0.02N.* Dilute 1.3ml of ACS reagent grade concd nitric acid to the mark with distilled water in a 100-ml volumetric flask

E) *Acid ferrous sulfate solution, 0.5N.* Dilute 8g of ACS reagent grade ferrous sulfate heptahydrate ($FeSO_4.7H_2O$) to the mark in a 1000-ml volumetric flask with distd water. Add three drops of concd sulfuric acid

Equipment:

a) Volumetric flasks, 1000ml and 100ml
b) Graduated cylinder, 250ml
c) Erlenmeyer flasks, 250ml, glass-stoppered
d) Ice bath

Sampling procedure. For taking a sample from its shipping container, attach to its valve the valve V_1 of tared sampler shown in Fig Et21. With valves V_1 and V_2 closed cool the sampler to 0°C(32°F) with ice. Open the container valve and then valve V_1 of the

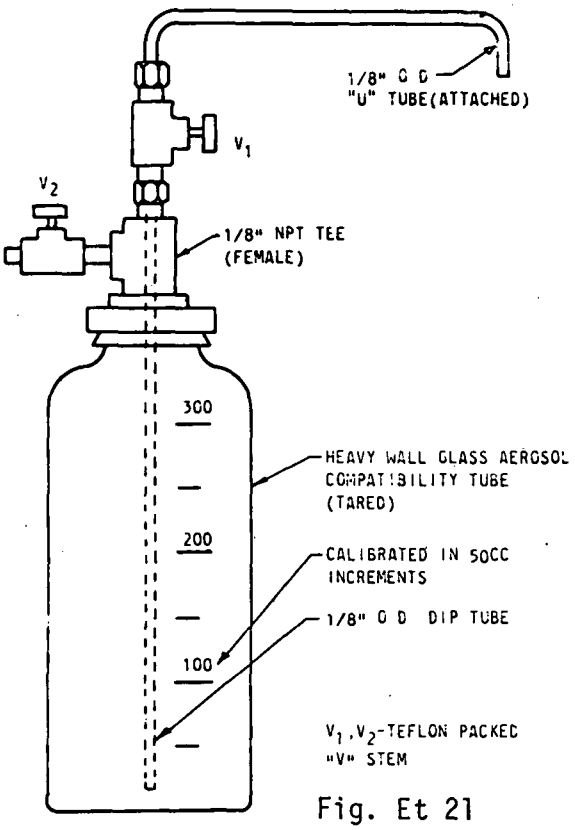


Fig. Et 21

sampler to allow the propellant to fill the sampler to the 300-ml mark. If an insufficient amt of liquid entered, open slowly valve V_2 until the 300-ml mark is reached. Then immediately close V_2 followed by closing V_1. Finally attach "U" tube, as shown in Fig Et21

Tests:

Examination of product. Examine the propellant contained in the sampler for clarity and suspended matter. The test method described in ASTM D1209-62 shall be used for the color measurement. The color shall not exceed the American Public Health Association color number 10

1) Ethylene oxide. Calculate the ethylene oxide concentration by the following formula:

Ethylene oxide, %Wt = 100.00 - %Wt (acidity + water + aldehyde + chloride + nonvolatile residue)

2) Acidity. The acidity shall be determined in accordance with ASTM D1613-64T with the following exception:

The propellant sample size shall be approximately 50 ml. Determine the weight taken by weighing the sampler to the nearest 0.1g before and after the liquid is removed

Calculate the results as follows:

Acidity as acetic acid, % Wt= $\frac{ml \times N \times 0.06005 \times 100}{W}$

where:

ml = milliliters of NaOH used

N = normality of NaOH

W = propellant sample weight titrated

3) Water. The water content shall be determined in accordance with ASTM E203-64, with the following exception:

Exception. The propellant sample size shall be approximately 50 ml. Determine the weight taken by weighing the sampler to the nearest 0.1g before and after the liquid is removed

4) Iron. The iron content shall be determined in accordance with D1068-62T, Method A, with the following exceptions:

a) The propellant sample size shall be approximately 100 ml. Determine the weight taken by weighing the sampler to the nearest 0.1g before and after the liquid is removed

b) Place the propellant sample in an evaporating dish and evaporate to dryness. Add 5 ml of concentrated HCl to the residue and evaporate the mixture to dryness. Swirl the mixture during evaporation to assure quantitative digestion of the residue. Quantitatively transfer the residue to a 100-ml volumetric flask with three 2-ml portions of 0.5N HCl. Proceed with the Method A analysis

c) An absorption cell with a 10-cm optical bath shall be used

Calculate the results as follows:

Iron, ppm/wt = $(W/W_s) \times 1000$

where:

W = milligrams of iron found

W_s = propellent sample wt in grams

5) Aldehyde. The aldehyde content of the propellant shall be determined in accordance with ASTM D2191-65 with the following exceptions:

a) The propellant sample size shall be approximately 50 ml. Determine the weight taken

by weighing the sampler to the nearest 0.1 g before and after the liquid is removed
b) Substitute a 12-oz heavy-wall aerosol compatibility tube with closure for the 500-ml glass-stoppered Erlenmeyer flask used for the titration
c) Substitute "W", the propellant sample weight in grams taken, for "W x M" in the formula for aldehyde content

6) Chloride. The chloride content of the propellant shall be determined in accordance with ASTM D512-62T, Method A, with the following exceptions:
a) The propellant sample size shall be approximately 50 ml. Determine the weight taken by weighing the sampler to the nearest 0.1 g before and after the liquid is removed.
b) Place the sample in an evaporating dish and add 5 ml of 0.25N NaOH. Evaporate the mixture to dryness and with distilled water quantitatively transfer the residue to a 250-ml Erlenmeyer flask. Proceed with the Method A analysis

7) Nonvolatile residue. The nonvolatile residue of the propellant shall be determined in accordance with ASTM D1353-64 with the following exception:
The propellant sample shall be approximately 100 ml. Determine the weight taken by weighing the sampler to the nearest 0.1 g before and after the liquid is removed

8) Acetylene. The acetylene content of the propellant shall be determined by the following procedure:
Transfer 150 ml of the silver nitrate reagent to each of two 250-ml glass-stoppered Erlenmeyer flasks. Place the flasks in a suitable ice bath until the temperature of the contents is 32° to 39.2°F (0° to 4°C). Add 7 to 9 drops of the mixed indicator to each flask and, if necessary, neutralize to a gray-green color with 0.02N sodium hydroxide if acid; or with 0.02N nitric acid if alkaline. Reserve one of the flasks for the blank and into the second flask pour 50 ml of propellant. Compare the color of the sample with that of the blank. A purple color in the sample solution indicates the presence of acetylene
Note: If a positive test is obtained, destroy the silver acetylide by adding an excess of acid ferrous sulfate solution to the sample flask before discarding the solution

Ethylene Ozonide (Äthylenozonid in Ger), $H_2C\text{-}O_3\text{-}CH_2$; mw 76.06, colorless, unstable, oily liq with pungent odor, sp gr 1.265 at 17.5/17.5°, N_D 1.4099 at 17.5°, bp 18° at 16mm; explodes violently on heating or by friction; decomposes explosively when treated with strong NaOH soln. Can be prepd by the action of purified ozone on a soln of ethylene in methylchloride at -70° in the complete absence of water (Refs 1 & 2). Ozonization of ethylene also was described by Briner & Schnorf (Ref 3)
Criegge (Ref 4) reported that one of the laboratory workers prepd and purified about 500 mg of ozonide using the method described in Refs 1 & 2 and then left the product standing till next day. On transferring it into another vessel a violent expln took place accompanied by a bright flame
This ozonide also is described by Tobolsky & Mesrobian (Ref 5)
Refs: 1)Beil 1, 184 & {633} 2)C. Harries & R.Koetschau, Ber **42**, 3305(1909) 3) E.Briner & P. Schnorf, Helv **12**, 154(1929) 4)R. Criegge, AgnewChem **65**, 399(1953) & CA **47**, 11737(1953) 5)Tobolsky & Mesrobian (1954), 180

Ethylene Pseudonitrosite, $O_2N.CH_2CH_2.NO.ON.CH_2CH_2NO_2$; mw 208.14, N26.92%, OB to CO_2-46.2%; ndls, mp 116-17°(dec), sol in concd H_2SO_4, insol in w, sl sol in alc. Prepd by passing ethylene and N_2O_4(Ref 2) or N_2O_3(Ref 4) through eth. The structure was first hypothesized in Ref 3, and later assumed in Ref 4, but there is no confirmatory evidence. Marshall (Ref 4) proposed its use as a secondary charge in blasting caps, eg, a base charge of 0.4-1.0 g ethylene pseudonitrosite over a priming charge of MF, etc, in a copper shell Ethylene pseudonitrosite is comparable in power and brisance to Tetryl
Refs: 1)Beil 1, 184 & (77) 2) N.J.Dem-

janow, Chem Zentr **1899** I, 1064 3) K. Ssidorenko, ibid **1914** I, 1069 4) J.Marshall, USP 1473825 (1924) & CA **18**, 588 (1924) 5)Blatt, OSRD **2014** (1944)

Ethylene Tetrafluoride or Tetrafluoroethylene (Perfluoroethylene), $F_2C{:}CF_2$; mw 100.02; col gas, fr p -142.5°, bp -78.4°; insol in w. Was prepd by Humiston (Refs 1&2) by treating charcoal with fluorine; carbon tetrafluoride was obtd at the same time. Accdg to Ref 3, it can be prepd by passing chlorodifluoromethane thru a hot tube. Serves as a raw material for prepn of polytetrafluoroethylene polymers (Ref 3)

Note: Accdg to Dr J.V.R.Kaufman, formerly of PicArsn, Minnesota Mining and Manufacturing Co of Minneapolis, Minn experienced in their laboratory an expln which was probably caused by sudden polymerization of Ethylene Tetrafluoride. This expln caused the loss of an arm of one of the investigators trying to purify the material by distillation

Refs: 1)Beil **1**, (77) 2)B.Humiston, JPhys Chem **23**, 573 (1919) 3)CondChemDict (1961), **1122-L** (Tetrafluoroethylene) 4)Sax, 3rd edit (1968), 1145-46

Ethylenetetranitramine or Tetranitraminoethylene, $(O_2N)_2N.H_2C.CH_2.N(NO_2)_2$; mw 240.09, N23.34% ($NO_2$), OB to CO_2 + 13.3%, liq. Prepd by reacting ethylenediamine dinitramine with nitronium fluoborate in acetonitrile at -30° (Ref 3). No props are given

Refs: 1)Beil, not found 2)ADL, Synthesis HE's, 2nd Report (1951), p 220 3)Aerojet General Corp, BritP1126591 (1968) & CA **70**, 67584 (1969)

Ethylene-iso-thiourea and Derivatives

Ethylene-iso-thiourea, 2-daminothiazolidine or 2-Amino-2-thiazoline

$SC(=NH)NHCH_2CH_2$ or $SC(NH_2)=NCH_2CH_2$; mw 102.16, ndls or flakes (from benz), mp 84-5°, sol in w, alc, chlf & warm benz. Prepd from 2-bromoethylamine hydrobromide and potassium rhodanide. There is a **picrate**, ndls(from w), mp 235° (dec)

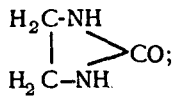
Refs: 1)Beil **27**, 136 & [194] 2)S.Gabriel, Ber **22**, 1141 (1889)

Nitroethylene-iso-thiourea, $C_3H_5N_3O_2S$; mw 147.15, yel ndls (from HAc), mp 203.4° (dec). Prepd by treating a suspension of the parent compd in benzene with nitrogen oxides. Structure not elucidated

Refs: 1)Beil **27**, 136 2)S.Gabriel & E.Leupold, Ber **31**, 2834 (1898)

Ethyleneurea and Derivatives

Ethyleneurea, Ethylenecarbamide, 2-Imidazolidine or 2,5-Diazacyclo-1-pentanone,



mw 86.09, wh ndls, mp 131-132.5°, sol in w and in hot alc, v sl sol in eth. Was first prepd by Fischer & Koch (Refs 1&2) by heating ethylenediamine with ethyl carbonate in a sealed tube. This method, as well as some later methods cited by Mulvaney & Evans (Ref 4), were uneconomical for industrial purposes. As ethyleneurea is an important chemical intermediate and has been used for prepn of expls such as EDNA, the Ordnance Dept of the US Army contracted in 1942 with Evans Research and Development Corp of New York City to devise an economical industrial method. As result of this contract a new general method for the synthesis of cyclic ureas was developed. This method involved the conversion of a mixture of ethylenediamine and carbon dioxide (or the addition compd ethylenediamine carbamate), to ethyleneurea under the influence of heat (temperature 200 to 230°) and pressure ranging from 400 to 900psi). Two processes, one a batch, the other continuous, were developed and carried thru a small pilot plant. Yields of 95% and better were obtd

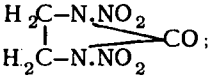
Bachmann et al(Ref 5) described a simple laboratory method of prepg ethyleneurea without using any sealed tube

Ethyleneurea gives on nitration the dinitrocompd

Refs; 1)Beil **24**, 2, (184) & [3] 2)E.Fischer & H.Koch, Ann **232**, 227 (1886) 3)G.C.Hale, USP2430874 (1947) & CA **43**, 691(1949)(Description of prepn from ammonium carbonate and an ethylene derivative such as ethylene-

diamine, ethylene oxide or ethyleneglycol) 4)J.F. Mulvaney & R.L.Evans, IEC **40**, 393-7 (1948) (15 refs) 5)W.E.Bachmann, etal, JACS **72**, 3133(1950) 6)CondChemDict (1971), 367

N,N'-Dinitroethyleneurea (DNEU); 2,5-Dinitro-2,5-diazacyclo-1-pentanone or 1-3,-Dinitro-2-imidazolidone



mw 176.09, N 15.91% (NO_2) OB to CO_2 -27.1%, wh prisms (from hot alc), density 1.796(cryst), 1.65(loading), mp 216-217° (decomp), explodes at higher temps, insol in water and in most org solvents, diffc sol in boiling alc, hydrolyzes to ethylenedinitramine on boiling with water. Was first prepd by Franchimont & Klobbie on treating ethyleneurea with concd nitric acid (Refs 1 & 2). Aaronson of PicArsn found that by using mixed nitric-sulfuric acid instead of straight nitric acid yields were increased from 53-60% to 92-95% of theory. In his laboratory procedure 1 part of powdered ethyleneurea was slowly sifted, with stirring, into 10 parts of mixed acid (HNO_3 22.0, H_2SO_4 68.5 & H_2O 9.5%) maintained during addition at below 10°. Stirring continued for 1-2 hours allowing the temp to rise but not above 30°. Then the slurry was filtered and ppt washed with water and dried. Methods of nitration were examined during WWII by the duPont Co (Ref 5) and it was concluded that nitration with straight strong nitric acid is preferable to nitration with mixed acid. Caesar & Goldfrank (Ref 7) patented a method of prepg DNEU by nitration at low temp of ethyleneurea dissolved in an inert solvent (such as $CHCl_3$) employing either nitrogen pentoxide or concd HNO_3. Yields of nearly 100% were claimed.

Properties of DNEU (Ref 6 & other sources)
Brisance - not detd
Detonation Velocity - 7690 m/sec at d 1.60 and 7890 at d 1.65, for unconfined charges 2.5 x 40 cm
Explosion Temperature - 235-240° in 5 seconds
Heat of Combustion - 388 kcal/mol
Heat of Formation - 31.2 kcal/mol
Hygroscopicity - gained 9.12% at RT & 100%RH in 24 hours
Impact Sensifivify - between those of TNT and Tetryl
Power by Ballistic Mortor - 129% TNT
Power by Trauzl Test - 120% TNT
Stability - fairly stable at below mp
Thermal Stability at 135° - not acid and no expln in 300 minutes
Vacuum Stability at 120° - 7.4cc gas evolved per 5g in 25 hours
Uses: As an intermediate for prepn of EDNA, described in this Vol as Ethylenedinatramine under Ethylenediamine. It is suitable for use as booster charges and as base charge in blasting caps and detonadors (Refs 4 & 6)
Refs: 1) Beil **24**,4 2) A.Franchimont & E.Klobbie, Rec **7**,16 & 243 (1888) 3)H.A.Aaronson,USP 2149260 (1939) 4) W.H.Rinkenbach & H.A.Aaronson, USP 2167679 (1939) & CA **33**,8992 (1939) 5) E.I. du Pont de Nemours, OSRD Rept **3466** (1944) 6) Blatt,OSRD Rept **2014** (1944) 7) G.Caesar & M.Goldfrank, USP 2400288 (1946) & CA **40**,4525 (1946)

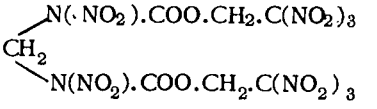
Ethylester of Benzoylbenzoic Acid. See Ethyl-o-benzoylbenzoate in this vol

Ethylester of Carbamic Acid or Urethane. See Carbamic Acid Ethylester in Vol 2 of Encycl, pp C40-R & C41-L

Ethylester of Dimethylaminocyanophosphoric Acid. See GA Chemical Warfare Agent described in Vol 2,p C167-R. Originally, it was German Agent of WWII called Tabun, Trilon 83, T83 or T100, described in PATR **2510** (1958),pGer 204-L

Ethylester of Trinitrocarbanilic Acid or Picrylurethane. See Vol 2,pC43-R

Ethylester (Trinitro Deriv) of N,N'- Dinitromethylonedicarbamic Acid,



mw 550.20, N 25.46%; crysts,mp 112°,

ignition temp 218°, d 1.89g/cc;impact sensitivity, ERL Machine with 2.5 kg wt & Type 12 tools = 10-20cm for 50% explns. It was prepd by nitration of Trinitroethanol Ester of Methylenedicarbamic Acid (qv) (Refs 1 & 2). Can be used as a solid ingredient in castable expls,analogous to use of RDX

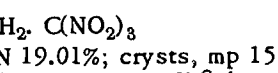
Refs; 1) M.B.Frankel & L.T.Carleton, Aerojet Engrg Corp Quarterly Progress Rept **589** (March 1952) (Contract N7 onr 46208) 2) W.F.Sager & D.V.Sickman, NAVORD Rept **483** (June 1952),p18

N-Ethylester (Trinitro Deriv) of Ethanesulfonamide,

$CH_3.CH_2.SO_2.NH.CH_2.C(NO_2)_3$; mw 272.20, N 20.58%; crysts,mp 171°,d 1.71 g/cc, ignition temp 281°; impact sensitivity, about that of Comp A or Torpex. It was prepd by addg twice the theoretical amt of Trinitroethanol at 100° to the sulfonamide (Refs 1 & 2). Can be used as a solid ingredient in castable expls, analogous to use of RDX

Refs: 1) R.H.Saunders et al, Hercules Powd Co Progress Rept (Oct 1949) (Contract Nord 9925) 2) W.F.Sager & D.V.Sickman, NAVORD Rept **483**(June 1952),p8

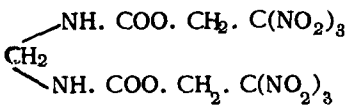
bis-[Ethylester (Trinitro Deriv)] of Fumaric Acid or bis (Trinitroethyl) Fumarate,



mw 442.18, N 19.01%; crysts, mp 152°, d1.72 g/cc, ignition temp 227°, impact sensitivity - about that of Pentolite. Prepd by treating the acid chloride with Trimitroethanol in the presence of $AlCl_3$ catalyst (Refs 1 & 2). Can be used as a solid ingrediant in castable expls, analogous to use of RDX

Refs: 1) Aerojet Engrg Corp Quarterly Progress Rept **386** (July 1949),p35 2) W.F. Sager & D.V. Sickman, NAVORD Rept **483** (June 1952),p11

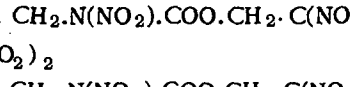
Ethylester (Trinitro Deriv) of Methylenedicarbamic Acid,



mw 460.19, N 24.35%; crysts, mp 123°; d1.70 g/cc, ignition temp 203°; sensitivity by impact using ERL Machine with 2.5 kg wt & Type 12 tools = 20-40cm. Produce by reaction of Trinitroethonol with Trinitroethyl Carbamate, or from methylenediisocyanate & Trinitroethanol (Refs). It can be used as a solid ingredient in castable expls, analogous to use of RDX

Refs: 1) Hercules Powd Co Progress Rept on Contract Nord 9925 (Aug 1950) 2) Aerojet Engrg Corp Quarterly Progress Rept **589** (March 1952)

Ethylester (Trinitro Deriv) of 2,5,5,8 - Tetranitro - 2,8 - diazanonanedioic Acid,



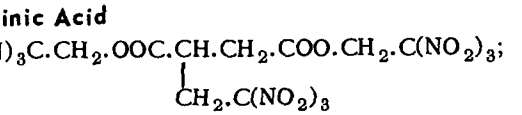
mw 696.31, N 24.14%; cryst, mp 149-50°, d 1.76 g/cc, hot bar ignition temp 211°, sensitivity to impact- about the same as PETN. It was prepd by nitrating with 100% nitric acid the bis (carbamate) resulting from reaction of 3,3-dinitropentane-1,5-diisocyanate & Trinitroethanal. Can be used as a solid ingredient in castable expls, analogous to use of RDX

Refs: 1) L.T. Carleton & M.B. Frankel, Aerojet - Genl Corp Rept **621** (1952) (Contract N7 onr 46208) 2) W.F.Sager & D.V.Sickman, NAVORD Rept **483** (June 1952),p23

Ethylester (Trinitro Deriv) of N,N,' 3,3 - Tetranitro - 1,5 - pentanedicarbamic Acid.

Same as above compd

bis-Trinitroethylester of Trinitroethylsuccinic Acid



mw 607.24, N 20.76%; crysts, d 1.75g/cc, hot bar ignition temp 210°; impact sensitivity approaches that of Tetryl. Prepd by esterification of Trinitroethanol with the adduct of nitroform & itaconic acid. The eutectic of this compd with TNETB melts below 65°

Ref: D.V.Sickman & W.F.Sager, NAVORD Rept **486** (1954)

Ethylethanolamine. See Ethylaminoethanol in this vol

Ethylethanolperoxide or Hydroxydiethyl Peroxide, $C_2H_5OOCHOHCH_3$; mw 106.12 liq, spgr 0.974 at 21.4/4°, bp 50-2° (50 mm Hg), n_D 1.4021 at 21.4°. Prepd by letting stand for one day a 5% eth soln of ethylhydroperoxide and acetaldehyde. If distilled in the presence of water, there is the danger of expln

Refs: 1) Beil, not found 2) A. Rieche, Ber **63B**, 2642–52 (1930) & CA **25**, 911 (1931) 3) G. J. Minkoff, ProcRoySoc **224A**, 176–91 (1954) & CA **49**, 57 (1955) (IR spectrum)

Ethylether or Ether. See Diethylether in Vol 5, p D1233 of Encyd. See also Ether in this Vol

Ethyl Ether (Analytical). Its estimation in mixtures with ethanol, water and acetone is described in this Vol under 'Ethanol Determination in Solvents Used in Manufacture of Smokeless Propellants' and also under 'Ethanol Determination in Nitrocellulose and Smokeless Propellants'

Ethylethoxyphenylurea and Derivatives

Ethylethoxyphenylurea (N-äthyl-N′-4-äthoxyphenyl-harnstoff in Ger), $C_2H_5NH\,CONHC_6H_4OC_2H_5$; mw 208.26 cryst (benz), mp 152°, sol in alc, acet & warm benz. Prepd by reacting p-phenetidine with ethylisocyanate in eth at 0°

Refs: 1) Beil **13**, [254] 2) H.F.J. Lorang, RecTravChim **46**, 641 (1927)

Nitroethyl - trinitroethoxyphenylurea [N-nitro-N-äthyl-N′-(2,3,6-trinitro-4-äthoxyphenyl)-harnstoff, in Ger], $C_2H_5N(NO_2)CONH[C_6H(NO_2)_3OC_2H_5]$; mw 388.25, N 14.43%, OB to CO_2 −74.2%, yel crysts, mp 92–8° (dec) sol in acet & benz. Prepd by treatment at −10 with 100% HNO_3, N-ethyl-N′-ethoxyphenylurea. Decomposition occurs on attempted recrystallization from acet, benz, chlf, or petr eth

Refs: 1) Beil **13**, [295] 2) H.F.J. Lorang, RecTravChim **46**, 642 (1927)

Ethyl-N-ethylcarbamate. See Ethylurethane

Ethylethylene. See Butene or Butylene in Vol 2 of Encycl, p B375-R

Ethylethyl . Hydrazine. See Dithylhydrazine in Vol 5 of Enycl, p D1237-L
Its Hexanitro Derivative is described as N,N′-Di(2,2,2-trinitroethyl) hydrazine or 1,2-Bis(2,2,2-trinitroethyl) hydrazine on p D1237-R in Vol 5

Ethylformal Polysulfide (LP–33). Used, under the code name Unit No 533, as fuel in some rocket propellants

Ref: PropIntManual SPIA/M2 (1962) (Conf)

Ethylformate, $HCOO.C_2H_5$; mw 74.08; col, flammable, pleasant smelling liquid, sp gr 0.9236 at 20/20° fr p -80.5°, bp 54.3°, fl p -20°C, n_D 1.35975 at 20°, vapar pressure 200mm at 20.6°; soly in w 11% at 18°; with gradual decompn; miscible with alc, ether & benz. Can be prepd by heating ethanol with formic acid in presence of sulfuric acid. Can be used as solvent for cellulose nitrate and acetate; as acetone substitute and as fumigant

Refs: 1) Beil **2**, 19, (16) & [26] J.W. Döbereiner, Ann **3**, 145 (1832) 3) CondChemDict (1961), 470-L

Ethylfuroate or Ethyl Pyromucate, $C_4H_3O.CO_2.C_2H_5$; mw 140.13; wh lfts or prisms, sp gr 1.1174 at 20.8/4°, mp 34°, bp 195° at 766 mmHg, insol in w; miscible with alc or ether. Can be prepd by saturating a soln of furoic acid in alc with HCl

Refs: 1) Beil **18**, 275 & [266] 2) H.Schwanert, Ann **116**, 267 (1860) 3) CondChemDict (1961), 470-R

Ethyl-β-furylacrylate (Furfuracrylate d'éthyl, in Fr), $C_4H_3O.CH{:}CH.COOC_2H_5$; mw 166.18, liquid sp gr 1.0891 (25/4°), mp 24.5°, bp 130° at 20 mm, 233-35° at 1 atm, n_D 1.5286 at 20° sol in alc & eth; insol in water. Was prepd by esterification of furfuracrylic acid, which in turn was prepd from furfural. Was tried in France as a possible substitute for centralite in solventless smokeless propints (poudre SD, in Fr), but found to be unsafiafactory

Refs: 1) Beil **18**, 300 2) R.Dalbert & H. Ficheroulle, MP **30**, 283-30 (1928)

Ethylglucopyranoside and Derivatives

Ethylglucopyranosides, $\underline{OCH(CH_2OH)CHOHCHOHCHOHCHO}C_2H_5$; mw 208.21, α-form: rh cryst (acet), mp 114.6° sol in w & hot alc; β-form: ndl (acet), mp 73°, sol in w & alc. Prepn of α-form by action of HCl on β-form, or by heating d-glucase in alc containing HCl; prepn of β-form by saponification of the tetraacetate; also enzymatic preps of both from d-glucose and alc

Refs: 1) Beil **31**, [188] 2) E.Fischer, Ber **28**, 1153 (1895) 3) W.Koenigs & E.Knorr, Ber **34**, 972 (1901) 4) E. Bourquelot, et al, JPharmChim [7] **7**, 149 (1913)

Ethylglucopyranoside Tetranitrate, $C_8H_{12}N_4O_{14}$; mw 388.20, N 14.43%, OB to CO_2 −33.0%, colorless ndls (alc), mp 99-100°, v sl sol in w. Prepd by reaction of ethylglucopyranoside with fuming nitric acid in acetic anhydride at 0° for one hour

Refs: 1) Beil, not found 2) D.M. Shepherd, JCS **1953**, 3635-37 & CA **49**, 873 (1955)

Ethylglycerate, $(HO)_2C_2H_3.CO_2.C_2H_5$; mw 134.13, liquid, sp gr 1.191 at 15/15°, bp 121° at 14 mmHg; sol in w and in alc. Can be prepd by heating glyceric acid with alc at 190°

Refs: 1) Beil **3**, 397, (141-2) & [264] 2) L.Henry, Ber **4**, 706 (1871)

NOTE: No azido or nitro derivs were found in the literature

Ethylglycinate and Derivatives

Ethylglycinate, $H_2N.CH_2.CO_2.C_2H_5$; mw 103.12, colorless oil, sp gr 1.028 at 20/4°, fr p <-20°, bp 148° at 748 mmHg, n_D 1.4242 at 20°; miscible with w, alc or eth. Can be prepd by treating glycine HCl in alc with HCl to get the HCl salt, then shaking an aq suspension of the salt with the proper amount of Ag_2O in eth **Nitrous acid salt** cryst (alc), dec 140°; sol in w, alc, chlf, eth, & benz. Prepd from HCl salt and silver nitrite; **picrate,** prisms (w), mp 155°

Refs: 1) Beil **4**, 340, (467) & [780] 2) T. Curtius, Ber **16**, 754 (1883)

N - Nitro - ethylglycinate (Nitraminoessigsäureäthylester in Ger), mw 148.12, N 18.91%, cryst (eth), mp 24-5°, n_D 1.4610 at 24°, sol in all organics. Prepd by nitrating N-carboxyethylethyl glycinate in the cold with nitric acid in eth to get the N-Nitro-Ncarboxyethyl compound, treating the latter with ammonia gas to get the N-nitro-N-ammonium salt, and finally acidifying with hydrogen sulfide. The **ammonium salt** melts with dec at 60°, is sol in alc, and is very hygroscopic

Refs: 1) Beil **4**, 575 2) A.Hantzsch & W.V.Metcalf, Ber **29**, 1683 (1896) 3) E.H.White & R.J.Baumgarten, JOC **29** (12), 3636-40 (1964)

Ethylglycinate Hydrochloride; Ethylglycine Hydrochloride, Ethylglycocoll Hydrochloride or Glycine Ethylester Hydrochloride, $H_2N.CH_2.CO_2.C_2H_5.HCl$; mw 139.58, wh to pale yel ndls, mp 140-144°, bp-sublimes; sol in w & alc; insol in hydrocarbons; decompd by alkalies. Can be prepd by treating glycine hydrochloride in absolute alcohol with HCl gas followed by heating. Used in synthesis and in rocket propellants

Refs: 1) Beil **4,** 342 2) T. Curtius & F.Göbel, JPrChem [2] **37,** 160 (1888) 3) CondChemDict (1961), 543-L(Glycine Ethylester Hydrochloride)

Ethylglycine Ethylester and Derivatives

Ethylglycine Ethylester or *Ethyl - 3 - azavalerate,* $CH_3CH_2NHCH_2CO_2C_2H_5$; mw 131.18, liq, bp 58°(16mmHg). Prepd by treating ethylglycine with HCl in alc

Refs: 1) Beil **4,** [787] 2) A.Skita & C. Wulff, Ann **453,** 205 (1926)

N-2,2-Dinitroethylglycine Ethylester or Ethyl- 3 - aza - 5,5- dinitrovalerate, $CH(NO_2)_2CH_2NHCH_2CO_2C_2H_5$; mw 221.17 N 12.66% ($NO_2$), yel plates (EtAc), mp 102-3° (dec). Prepd from the reaction of the soduim salt of 2,2 - dinitro-ethanol with ethylglycinate hydrochloride

Refs: Beil, not found 2) H. Feuer, et al JACS **76,** 5124-6 (1954) & CA **49,** 13091-2 (1955)

N-2,2-Dinitroethyl - N - nitroglycine Ethylester or **Ethyl - 3 - aza - 3,5,5-trinitrovalerate,** $CH(NO_2)_2CH_2N(NO_2)CH_2CO_2C_2H_5$; mw 266.17, N 15.79% ($NO_2$) OB to CO_2 −54.1%, crysts(CCl_4), mp 41.2°. Prepd by treating the 5,5 - dinitro compound with HNO_3/H_2SO_4 at 0°, then raising temp to 55–60°. Treatment with $NaOCH_3$ in methanol give the **5-sodium salt,** lt yel crysts dec at 87-8°

Refs: 1) Beil, not found 2) H. Feuer, et al, JACS **76,** 5124-6 (1954) & CA **49,** 13091-2 (1955)

Ethylglycocoll Hydrochloride. See Ethylglycinate hydrochloride in this vol

Ethylglycolate and Derivatives

Ethylglycolate, $HOCH_2.CO_2.C_2H_5$; mw 104.10, colorless liq, sp gr 1.087 at 15/4°, bp 160°, v sol in alc & eth. It can be prepd by heating at 150° α-chloroethylacetate, sodium acetate and alc

Refs: 1) Beil **3,** 236,(91) & [171] 2) W.Heintz, Ann **123,** 326 (1862)

Ethylglycolate Nitrate, $O_2NO.CH_2CO_2C_2H_5$; mw 149.10, OB to CO_2 - 69.8%; col liq bp 180-2°, 75-7°(20mm Hg), sp gr 1.249 at 20°, n_D 1.4178 at 20°. Prepd from ethylglycolate and HNO_3/H_2SO_4 (Ref 2) or more recently by using HNO_3 (HNO_3/Ac_2O) or 70/30 HNO_3/H_2SO_4 with α-I or α-Br-ethyl acetate at low temp (Ref 3). The compd expl on heating

Refs: 1) Beil **3,** 238 2) L.Henry, AnnChim [4] **28,** 424(1873) 3) N.V.Svetlakov, et al, ZhOrgKhim **4**(12), 2096–9 (1968) & CA **70,** 67507 (1969)

Trinitroethylglycolate Nitrate, (O_2NO)-$CH_2.COO.CH_2.C(NO_2)_3$; mw 284.10, N 19.73%; OB to CO_2 + 5.6%. There is no mention of this material in the open literature. Presumably prepn was by esterification of the free acid nitrate (which is known) with 2,2,2-trinitroethanol

Refs: 1) Beil, not found 2) USRubber Co Quarterly Rept No **25** on Contract Nos 10129 & 12663, Nov 1, 1953-Feb 1, 1954, pp 3 & 7

Ethylglycol Ether and Derivatives

Ethylglycol Ether, $HOCH_2CH_2OC_2H_5$; mw 90.12, colorless liq, sp gr 0.935 at 15/15°, bp 134-5°(48 mm Hg), sol in liq NH_3. Prepd by heating glycol, ethyl iodide and sodium

Refs: 1) Beil **1,** 467,(244) & [518] 2) M.H.Palomea, Ber **42,** 3876 (1909)

Ethylglycol Ether Nitrate $O_2NO\ CH_2CH_2OC_2H_5$; mw 135.12, liq, sp gr

1.1118 at 20°, bp 64°(12 mmHg), n_D 1.4132 at 22°. Prepd by treating ethylglycol ether with HNO_3/Ac_2O under 5°(Ref 2)

Refs: 1) Beil, not found 2) G. Desseigne, BullSocChim **1946**, 98-9 & CA **41**, 2001 (1947) 3) R.A.G.Carrington, SpectrochimActa **16**, 1279-93 (1960) & CA **55**, 12035 (1961) (Infrared spectrum) 4) J.Taylor & S.M.Budd, USP 2966404 (1960) & CA **55**, 12856 (1961) (Use in a gas - generating compn)

Mononitroethylglycol Ether or Nitroethoxyethenol

$HOCH_2CH_2OCH_2CH_2NO_2$; mw 135.12, liq, sp gr 1.2185 at 20°, bp 92°(3 mmHg), n_D 1.4516 at 20°. Prepd by heating 2 - nitroethanol with ethylene oxide for 6 hours at 80° in the presence of sulfuric acid

Refs; 1) Beil, not found 2) M.S.Malinovskii, et al, UkrKhimZh **34**(5), 489–91 (1968) & CA **69**, 58779 (1968)

Ethylglycolic Acid, Ethoxyacetic Acid or Glycolic Ethylether

$C_2H_5.O.CH_2.CO_2H$; mw 104.10; colorles liq sp gr 1.102 at 20/4°, bp 206-7°, 128-30° (40 mmHg), n_D 1.4194 at 20°. Can be prepd by mixing chloroacetic acid and sodium ethoxide in alc

Refs: 1) Beil **3**, 233, (89) [170] 2) M.Sommelet, AnnChim [8] **9**, 489 (1906)

NOTE: No azido or nitrated derivs were found in the literature

Ethylguanidine and Derivatives

Ethylguanidine

$C_2H_5.NH.C(:NH).NH_2$; mw 87.125, N 48.23%; mp 210-12° (Ref 5). Prepd as various salts by reacting $H_2NC(=NH)SCH_3.HI$ with 30% alcoholic ethylamine or $C_2H_5NHC(=NH)SCH_3.HI$ with 25% ammonia; prepd as free base (apparently) by reacting $H_2NC(=NH)SCH_3$ with $EtNH_2$ in NH_4OH (Ref 5). Heating ethylamine nitrate with $CaCN_2$ or NCNH (=NH) NH_2 gives the **nitrate salt**, mp 108-9°(Ref 3). Sulfate salts are obtained directly by heating $H_2NC(=NH)OEt.EtHSO_4$ with ethylamine for 3 hours at 60°(Ref 4); **picrate**, mp 178–80° (Ref 2)

Refs: 1) Beil **4**, [609] 2) M.Schenck & H.Kirchof, H-S' sZPhysiolChem **154**, 295-7 (1926) & CA **20**, 3284 (1926) 3) P.E.Gagnon et al, CanJChem **36**, 1436-40 (1958) & CA **53**, 9054 (1959) 4) E.Roberts, BritP 817749 (1959) & CA **54**, 9775 (1960) 5) R.Pant, H-S' s-ZPhysiolChem **335**, (2), 272-4 (1964) & CA **60**, 13311 (1964)

1-Ethyl-3-nitrosoguanidine

$C_2H_5NHC(:NH)NHNO$; mw 116.12, N 48.25% Prepd as the $C_6H_{14}N_8NiO_2$ salt, bright red powder, expl. Prepd by addition of $Ni(NO_3)_2$ and NaAc in w to the product from the Raney nickel/H_2 reduction of 1-ethyl-3-nitroguanidine(see below) (120 atm H_2, ambient temp, CH_3OH solvent). The reduction product itself is extremely sol in w and alc, but does not recrystallize in a pure state from either

Refs: 1) Beil, not found 2) E.Lieber & G.B.L.Smith, JACS **59**, 2283-7 (1937) & CA **32**, 502 (1938)

1-Ethyl-3-nitroguanidine

$C_2H_5NHC(:NH)NHNO_2$; mw 132.12, N 42.41% cubes, mp 147-8°, sol in alc, cold w & eth. Prepd from nitroguanidine and 10% aqueous ethylamine at 60-70°(Ref 2) or by treating the nitrate salt of ethylguanidine with sulfuric acid for 3 hours at 5°(Ref 3)

Refs: 1) Beil **4**, 609 2) T.L.Davis & S.B.Luce, JACS **49**, 2304 (1927) & CA **21**, 3348 (1927) 3) P.E.Gagnon, et al CanJChem **36**, 1436-40 (1958) & CA **53**, 9054 (1959)

1-Ethyl-1-nitroso-3-nitro-guanidine

$C_2H_5N(NO)C(:NH)NHNO_2$; mw 161.12, N 43.46% OB to CO_2 -64.6%, mp 114.5°(dec) (118°dec). Prepd by treating 1-ethyl-3-nitro-guanidine with nitrous acid

Refs: 1) Beil, not found 2) A.F.McKay JACS **71**, 1968-70 (1949) & CA **43**, 9035 (1949)

1,1-Dinitro-2-guanidino-ethane,
$CH(NO_2)_2CH_2NHC(=NH)NH_2$;mw 177.12, N 39.54%,OB to CO_2 - 49.7%,gold plates (w),mp 196-9°. Prepd by treating 1,1,1-Trinitroethane at 0-5° with guanidinium carbonate which has been reacted with NaOEt in alc
Refs: 1) Beil,not found 2) L. Zeldin & H.Shechter,JACS **79**, 4708-16 (1957) & CA **52**, 2735 (1958)

1-Nitro-3-(2,2,2-trinitroethyl)-guanidine or Tetranitro-ethylguanidine
$O_2N.NH.C(:NH).NH.CH_2.C(NO_2)_3$;mw 267.115,N 20.97% (NO_2), OB to CO_2 - 3.0%, colorless crysts mp 164° (168-170°),bp-ignites at 200°. Can be prepd by the reaction of nitroform with methylolnitroguanidine. (Refs 2-4). Other method is given in Ref 5,p61. It is an expl with sensitivity about that of Tetryl and Vacuum stability at 100°-11 ml of gas per gram evolued in 48 hours. It may be nitrated to Pentanitroethylguanidine
Refs: 1) Beil,not found 2) USRubberCo, Progress Report **No5** (1948-1949),p30 3) Ditto,Progress Report **No6** (1949), pp4-6 4) Ditto,Progress Report **No8** (1949),pp23-4 5) ADL, Synthesis HE's, 2nd Report (1951) pp53 & 61 6) W.F.Sager & D.V.Sickman, NAVORD Report **483** (1952),p33. 'Research and Development in NewChemicalHighExplosives' 7)H.A.Hageman,USP3035094 (1962,applied 1949) & CA **57**, 8792 (1962)

1,3-Dinitro-3-(2,2,2-trinitroethyl)-guanidine or Pentanitroethylguanidine
$O_2N.NH.C(:NH).N(NO_2).CH_2.C(NO_2)_3$,mw 312.11, N 22.43%,($NO_2$),OB to CO_2 + 10.3%,colorless crysts,mp ? . Can be prepd by nitrating 1-Nitro-3(2,2,2-trinotroethyl)-guanidine. The compound is not mentioned in the open literature
Ref: 1) Beil,not found 2) USRubberCO-ProgressReport **No9** (1950),pp19-21 3) ADL,SynthesisHE's (1951)

Ethylhexahydro Pyrimidine and Derivatives
5-Ethylhexahydro Pyrimidine
$C(C_2H_5)=CHNH=CHNH=CH$;mw 114.19. This compound is not reported in the literature,nor is there an unsaturated version
Refs: 1) Beil,not found 2) CA,not found

5-Ethylhexahydro-5-nitro-pyrimidine
$C(C_2H_5NO_2)CH_2N(NO)CH_2N(NO)CH_2$; mw 159.19 mp 159-60°. Prepd by heating for H/2 hours at 95° a 1/3/3mole ratio of nitropropane/formaldehyde/ammonia
Refs: 1) Beil,not found 2) T.Urbański et al, RocznikiChem **28**, 169-73 (1954) & CA **49**, 8826 (1955)

5-Ethylhexahydro-5-nitro-1,3-dinitroso Pyrimidine
$C(C_2H_5NO_2)CH_2N(NO)CH_2N(NO)CH_2$; mw 217.19,N 19.34% ($NO_2$),mp 116°. Prepd by treating with 20% $NaNO_2$ the hydrochloride salt of 5-nitro-2-ethylhexahydropyrimidine
Refs: 1) Beil,not found 2) T.Urbański etal,RocznikiChem **28**, 169-73 (1954) & CA **49**, 8826 (1955)

Ethylhexahydro-triazine and Derivatives
1-Ethyl-2,4,6-hexahydro-1,3,5-triazine,
$(C_2H_5)N.CH_2.NH.CH_2.NH.CH_2$: mw 115.18. This compound is not reported in the literature,nor is there an unsaturated version
Refs: 1) Beil,not found 2) CA,not found

3,5-Dinitro-1-ethyl-2,4,6-hexahydro-1,3,5-triazine
$(C_2H_5)N.CH_2.N(NO_2).CH_2.N(NO_2).CH_2$; mw 205.17, N 13.65% $(NO_2)_3$ wh crysts,mp 88-89° (Ref 2),96-7° (dec,Ref 3). Can be prepd from methyldinitramine, 40% aq formaldehyde and ethylamine at 0°
Refs: 1) Beil,not found 2) F.Chapman et al,JCS **1949**, 1639-40 & CA **44**, 1412 (1950) 3) R.Reed Jr.,JACS **80**, 439-44 (1958) & CA **52**, 10110 (1958)

2-Ethyl-1,3-hexanediol,2-Ethyl-3-propyl-1,3-propanediol or Ethohexadiol
$C_3H_7.CH(OH).CH(C_2H_5).CH_2OH$; mw 146.23, nearly colorless,odorless,somewhat viscous, hygroscopic liquid irritating to eyes,sp gr 0.9422 at 20/20°,fr p-sets to a glass at -50°,

bp 244° at 760 mm, 133° at 15 mm, n_D 1.4511 at 20°, viscosity (absol) 323 cps, vapor pressure <0.01mm at 20° (Refs 4 & 5); fl p (Cleveland open cup) 265°F; sol in alc & in eth; soly in w. Was first prepd in 1928 by Grignard & Fluchaire (Ref 2 & 4), in two steps: 1) Condensation of butyraldehyde with ethoxy magnesian iodide to produce 2-ethyl-1,3-hexanediol dibutyrate and 2) Saponification of this diester. In 1943 Kulpinski & Nord (Ref 3 & 4) prepd 2-ethyl-1,3-hexanediol by hydrolyzing its monobutyrate, which was previously obtd by the reaction of butyraldehyde with magnesium aluminum butoxide. Carbide & Carbon Co produced it commercially by the hydrogenation of butyraldol (Ref 4, p 286)

It is severely toxic when taken orally, but can be applied to human skin without irritation or sensitization (Ref 4, p 323-24). High purity 2-ethyl-1,3-hexanediol is considered as one of the best insect repellants. It was used in great quantities during WWII against mosquitoes, black flies gnats, chiggers, and other biting insects (Ref 4, p 286); it has also been used as a cosmetic ingredient and as a vehicle and solvent in formulation of printing inks (Ref 5). No info about its use in Indochina. Typical specification for commercial product and tests are given in Ref 4, pp 346-50

Refs: 1) Beil **1**, [556] 2) V.Grignard & M.Fluchaire, AnnChim (10) **9**, 5-54 (1928) 3) M.S.Kulpinski & F.F.Nord, JOrgChem **8**, 256-70 (1943) 4) Curme & Johnston, "Glycols" (1952), 286, 323-24 & 346-50 5) CondChemDict (1961), 470-R 6) Sax, 3rd edit (1968), p 752-R

2-Ethylhexenal. See 2-Ethyl-3-propylacrolein

Ethylhexoic Acid and Derivatives

2-Ethylhexoic Acid

$C_4H_9.CH(C_2H_5).COOH$; mw 144.22, mild-odored liq, sp gr 0.9077 at 20/20° C, fr p -83°, bp 226.9°, vapor pressure 0.03mm at 20°, sl sol in w. Can be prepd by oxidizing the corresponding aldehyde. Used as a pesticide and herbicide. Its metallic salts, especially of Pb, Mn, Co & Zn, are used as paint and varnish driers; its salts of light metals such as of Li, Mg, Ca & Al can convert some mineral oils to greases. Al salt is an excellent gelling agent for liquid hydrocarbons, such as gasoline and other petroleum fractions. High mol wt esters of this acid are used as plasticizers

Refs: 1) Beil **2**, 349 & [304] 2) J.A. Raupenstrauch, Monatsh **8**, 115 (1887) 3) CondChemDict (1961), 471-L 4) Sax, 3rd edit (1968), p 753-L

Lead 2-ethylhexoate. has been used in rocket propellants Unit Nos 383 & 387. Their compositions are listed in conf Propellant Manual SPIA/M2 (1962)

2-Ethylhexyl Acetate or Octyl Acetate

$CH_3.COO.CH_2CH:(C_2H_5)(C_4H_9)$; mw 172.26; colorless stable liq, sp gr 0.873 at 20/20°, fr p -93°, bp 199°, 95° at 25mm fl p 180° F, n_D 1.4204 at 20° vapor pressure 0.4mm at 20°, viscosity 0.0154 poise at 20°; sl sol in w, miscible with alc. Can be prepd by reacting 2-ethylhexanol with acetyl chloride. Used as solvent for NC, resins, lacquers and baking finishes

Refs: 1) Beil, not found 2) A.J.VanPelt, Jr. & J.P.Wibaut, RecTravChim **60**, 55-64 (1941) & CA **35**, 5090 (1941) 3) CondChem Dict (1961), 471-L & 471-R 4) Sax, 3rd edit (1968), p 753-L

2-Ethylhexyl Acrylate

$CH_2:CH.COO.CH_2.CH(C_2H_5).C_4H_9$; mw 184.28, colorless liq with pleasant odor sp gr 0.8869, fr p -sets to glass at - 90°, fl p 180° F, bp 85° (8mm Hg), n_D 1.4365 at 20° vapor pressure 0.1mm at 20°; insol in w; sol in org solvents. It can be prepd by treatment of methyl acrylate with 2-ethylhexanol. Used for plastics, protective coatings and water - based paints. Rohn & Haas Co proposed it as fuel-binder in some propellants, such as Unit No 600 described in conf Propellant Manual SPIA/M2 (1962)

Refs: 1) Beil, not found 2) C.E.Rehberg, et al, JACS **66**, 1723-4 (1944) & CA **39**, 907 (1945) 3) CondChemDict (1961), 471-R 4) Sax, 3rd edit (1968), p 753-R

2-Ethylhexyl Alcohol,2-Ethylhexanol or Octyl Alcohol
$CH_3(CH_2)_3.CH(C_2H_5).CH_2OH$; mw 130.22;colorless,sl viscous liq,sp gr 0.83 at 20/20°, fr p>-76°, bp 183.5°, n_D 1.4300 at 20°, fl p 81°C,vapor press 0.36mm at 20°; sl sol in w; miscible with org solvents. Can be prepd by aldolization of acetaldehyde or of butylaldehyde, followed by hydrogenation. Used in solvent mixts for NC,paints,lacquers; as defoaming & wetting agent and in organic synthesis (Ref 3)
Refs: 1) Beil **1**,[453] 2) M.Guerbet,CR **133**, 1222 (1901) 3) CondChemDict (1961) 471-R 4) Sax,3rd edit (1968), p 753-R

Ethyl Hydracrylate and Derivatives
Ethyl Hydracrylate
$HO.CH_2.CH_2.COOC_2H_5$; mw 118.13; colorless liq,sp gr 1.064 at 25,bp 185-90°,81°(13mm Hg), n_D 1.4271 at 23°, miscible with w,alc & eth. Can be prepd by condensing polyoxymethylene with ethylbromoacetate and zinc in half and half eth and EtAc
Refs: 1) Beil **3**, 297,(113) & [213] 2) E.E. Blaise & M.Maire,Bull SocCh [4] **3**, 266 (1908) 3) CondChemDict (1961) - not found

Ethyl 2- Nitrohydracrylate
$HOCH_2CH(NO_2)CO_2C_2H_5$; mw 163.13,sp gr 1.2662 at 20°,bp 102-3°(2mm Hg), n_D 1.4482 at 20°, dec on heating. Prepd by reacting formaldehyde with ethyl nitroacetate and NaAc at -15°
Refs: 1) Beil,not found 2) K.K. Babievskii, et al, DoklAkadNaukSSSR **160**, (1),103-5 (1965) & CA **62**, 11702 (1965) 3) See also CA **69**, 105825(1968) for a sodium deriv

Ethyl Nitratohydracrylate
$O_2NOCH_2CH_2CO_2C_2H_5$; mw 163.13,sp gr 1.262 at 20°, bp 92-3°(11mm Hg), 60-4°(1.5mm Hg), n_D 1.4258 at 25°, expl on dist at 760mm Hg. Prepd by electrolysis of a mixture of KNO_3, and potassium ethylsuccinate (Ref 2), or by treating $AgNO_3$ with ethyl 3-bromopropionate in acetonitrile (Ref 3)
Refs: 1) Beil,not found 2) Fr.Fichter & W.Steinbuch, HelvChimActa **26**, 695-704 (1943) & CA **37**, 5699 (1943) 3) A.F.Ferris et al, JACS **75**, 4078 (1953) & CA **49**, 8157 (1955)

Ethyl Hydrazine and Derivatives
Ethyl Hydrazine
$C_2H_5.NH.NH_2$; mw 60.10, N 46.61%,hygr liq, bp 101°; very sol in w,alc or eth. Can be prepd by reducing ethylnitramine with zinc and HCl
Refs: 1) Beil **4**, 550,(561) & [959] 2) J.Thiele & C.Meyer,Ber **29**, 963 (1896)

2,2-Dinitroethyl Hydrazine
$(O_2N)_2CHCH_2NHNH_2$; mw 150.095, N 37.33%, OB to CO_2 -32.0%, mp 137-140°, sp gr 1.679,sol in dimethyl sulfoxide & hot w. Prepd by reacting 2,2-dinitro-1,3-propanediol with hydrazine acetate in alc. The Olin-Mathieson Drop Wt Test value for impact sensitivity is 21 kg cm.
Refs: 1) Beil,not found 2) J.M.Greendorfer, USP3314997 (1967) & CA **67**,73135 (1967)

Ethyl Hydride. Same as Ethane

Ethylhydroperoxide and Derivatives
Ethylhydroperoxide
$C_2H_5.O_2H$; mw 62.07, colorless liq,fr p about -100°,bp 41-42° at 55mm Hg,n_D 1.3608 at 20°, miscible with w, alc or eth. Was first prepd in 1901, together with diethylperoxide, by Baeyer & Villiger(Refs 1 & 2) from diethylsulfate and alkaline soln of hydrogen peroxide (See also Ref 3). Can also be prepd by treating ethyl chloride, ethyl sulfate or ethanol with H_2O_2 in presence of strong acids (See also Refs 4,5,6,& 7). It is a strong oxidizing agent and easily explodes on heating or on impact, even when in soln

Ethylhydroperoxide Salts. Several salts are known,some of them explosive. Its *Barium Salt*,$Ba(OO.C_2H_5)_2+2H_2O$, consists of prisms

easily sol in w and insol in alc. Explodes on heating

Refs: 1) Beil **1**, 323 & [324] 2) A.Baeyer & V.Villiger, Ber **34**, 738 (1901) 3) A.Rieche & F.Hitz, Ber **62**, 245 (1929) 4) E.J.Harris, ProcRoySoc **A173**, 126 (1939) 5) N.Milas, USP 2176407 (1939) 6) A.C.Egerton et al, " Faraday Society Discussion on Hydrocarbons" **10**, 278 (1951) 7) Tobolsky & Mesrobian (1954), pp2, 158 & 177

Ethyl Hydroxyacetamide

$HO.CH_2.CO.NH.C_2H_5$; mw 103.12, N 13.58%; oily liq, bp 127-129° at 2.5mm Hg, sol in w, alc & eth. Was prepd by mixing ethyl chloroacetate and alc ethylamine in the cold

Note: Attempts to nitrate it by dissolving 2g of oil in 10ml of white nitric acid, heating the soln to 50–60° for 30mins and then pouring into ice water, produced an oil. This oil gradually disappeared on standing, probably due to hydrolysis

Refs; 1) Beil **4**, 125 2) W.Heintz, Ann **129**, 29 (1864)

Ethylhydroxyaniline. See N-Ethylaminophenol in this Vol

2-Ethylhydroxy-*iso*-butyrate

$(CH_3)_2C(OH)CO_2.C_2H_5$; mw 132.16, colorless liq, sp gr 0.978-0.986 at 20°, fr p?, bp 149-150° sol in w, alc or eth; decompd by hot w. Can be prepd by heating ethyl iodide with the potassium salt of α-hydroxy-iso-butyric acid at 120-130°. Used as a solvent for NC & cellulose acetate and in organic synthesis.

Refs: 1) Beil **3**, 315, (120) & [223] 2) R.Fittig & L.Paul, Ann **188**, 54 (1877) 3) CondChemDict (1961), 472-L

Ethyl-1-hydroxyethyl Peroxide

$H_3C.CH(OH)-OO-C_2H_5$; mw 106.12, oily liq, sp gr 0.974 at 21.4°, bp 50-52° at 50mm Hg, n_D 1.4021 at 21°. Can be prepd by mixing acetaldahyde and ethylhydroperoxide in eth

Refs: 1) Beil, not found 2) A.Rieche & F.Hitz, Ber **63**, 2642 (1930) & CA **25**, 911 (1931) 3) Tobolsky & Mesrobian (1954), 173 4) G.J.Minkoff, ProcRoySoc (London) **A224**, 176–91 (1954) & CA **49**, 57 (1955) (Infrared spectrum)

Ethylhydroxymethyl Peroxide

$CH_2(OH)-OO-C_2H_5$; mw 92.09, oily liq, sp gr 1.045 at 16/4°, bp 46-48° at 13mm, n_D 1.404 at 16°; sol in w, decompg on standing; very sol in alc & in eth; sol in benz. Can be prepd by interaction of equivalent quantities of ethylhydroperoxide and formaldehyde in ethereal soln. Puffs off weakly on heating

Refs: 1) Beil, not found 2) A.Rieche & F.Hitz, Ber **63**, 2646 (1930) & **A 25**, 911 (1931) 3) Tobolsky & Mesrobian (1954) 173

Ethylhyroxymethyl-propanediol and Derivatives

2-Ethyl-2-(hydroxymethyl)-1,3-propanediol or 2,2--Di(hydroxymethyl)-butanol

$C_2H_5C(CH_2OH)_2CH_2OH$; mw 134.18, wh plates hyg, mp 58.8-59°, bp 160° (5mm Hg), very sol in w & alc, sol in acet. Prepd from butyraldehyde and formaldehyde in CA $(OH)_2$ solution. Used extensively to make polyurethane resin foams

Refs: 1) Beil, not found 2) I.G.Farbenindustrie A.-G., BritP 484619 (1938) & CA **32**, 7609 (1938) 3) K & O, Vol 1 (1963), 595

2-Ethyl-2 (hydroxymethyl)-1,3-propanediol Dinitrate

$C_2H_5C(CH_2ONO_2)_2CH_2OH$; mw 224.17, N 12.50%, OB to CO_2 -78.6%. Prepd in 88% yield from 2-ethyl-2(hydroxymethyl)-1,3-propanediol, urea, 1,3-dichloropropane, 75% nitric acid and 75% sulfuric acid at –15°.

No props are given

Refs: 1) Beil, not found 2) F.Krupp, Ger P 1160427 (1964) & CA **60**, 9150 (1964)

2-Ethyl-2(hydroxymethyl)-1,3-propanediol Trinitrate or 2,2-Di(hydroxymethyl)-butanol Trinitrate,

$C_2H_5C(CH_2ONO_2)_2CH_2ONO_2$; mw 269.17, N 15.61%, OB to CO_2 -50-5%, wh powd, mp 51°, sp gr 1.48-0.50(cast), sol in acet. Prepd by nitration of 2-ethyl-2-(hydroxymethyl)-1,3-propanediol (Ref 3,6). **Detonation velocity** in 3-cm diameter cartridge at 1.48-1.50 g/cc 6300m/s cast, 7050 m/s compressed; expln temp is 220° (compared with 215° for PETN and 210° for NG in same apparatus), **heat of comb** at C_v is 829.2 kcal/m; **impact sensitivity** is 3.6 kgm; **power** by Pb block 115.5%.

Refs: 1) Beil, not found 2) H.Henkin & R.McGill, IndEngChem **44**, 1391-5 (1952) & CA **46**, 8857 (1952) (Expln temp) 3) L. L.Médard, MP **35**, 113-15 (1953) & CA **49**, 6082 (1955) 4) L. Médard & M. Thomas, MP **35**, 155–73 (1953) & CA **49**, 11284 (1955) 5) L. Médard, MP **36**, 8792 (1954) & CA **50**, 6793 (1956) (Expl props) 6) G. Bourjol, MP **36**, 79–85 (1954) & CA **51**, 10371 (1957)

Ethylhypochlorite, $C_2H_5.O.Cl$; mw 80.52, yel volatile liquid, sp gr 1.013 at -6/4°, bp 36° at 752mm; explodes at higher temp, violently explodes by action of direct sunlight; miscible with ether, benz & chlf. Can be prepd by the action of chlorine on ethanol (precooled to -18°) in presence of aq soln of NaOH. It is unstable

Refs: 1)Beil **1**, 324, (164) & [325] 2) T.Sandmeyer, Ber **18**, 1768 (1885)

Ethylhyponitrite. See Diethylhyponitrite in Vol 5 of Encycl, pD1238-L

Ethylidene Acetobenzoate and Derivatives

Ethylidene Acetobenzoate or Ethylidine Benzoacetate (Benzoat des Acetoins in Ger) $C_6H_5.CO_2.CH(COCH_3).CH_3$, mw 192.22, cedar-scented liq, sp gr 1.104 at 18°, bp 140-1° (8mm Hg). Prepd from acetoin and benzoylchloride in pyridine. Can be prepd commercially by interaction of benzoic acid and vinyl acetate in presence of catalysts. Used as solvent for NC, cellulose acetate and natural & synthetic resins

Refs: 1) Beil **9**, 149 2)O.Diels & E.Stephen, Ber **40**, 4340 (1907) 3)CondChemDict (1961), 472-L

Ethylidene Aceto-3,5-dinitrobenzoate, $C_6H_3[3.5\text{-}(NO_2)_2]\ CO_2CH(COCH_3)CH_3$; mw 282.21. Presumably prepd from acetoin and "3,5-dinitrobenzoic acid", no props given

Refs: 1)Beil, not found 2)K.Maček et al, Collection CzechChemCommun **29**, 311-15 (1964) & CA **60**, 6216(1964)

Ethylidene Acetone, Methylpropenyl Ketone or 3-Pentene-2-one, $CH_3.CH{:}CH.\text{-}CO.CH_3$; mw 84.11, col liq, sp gr 0.856 at 20°, bp 122-124° at 745mm, sol in w. Can be prepd by heating pentanol-4-2-one at 250°

*Refs: 1)*Beil **1**, 732,(381) & [791] 2)L.Claisen, Ber **25**, 3166(1892) 3)Sax, 3rd edit (1968), p 999-R (3-Pentene-2-one)

Ethylideneaminoguanidine and Derivatives

1-Ethylideneaminoguanidine, $CH_3CH{:}N.NHC(=NH)NH_2$; mw 100.12, N55.96%. Prepd from aminoguanidine and acetaldehyde in the usual way of making a hydrazone; no props given

Refs: 1)Beil, not found 2)P.Grammatikatis, BullSocChimFr **1952**, 446-538 & CA **47**, 3817(1953)

Ethylideneaminoguanidine Nitrate

$CH_3CH{:}N.NHC(=NH)NH_2.HNO_3$; mw 163.14, N 42.93%, OB to CO_2-73.6%, pr (alc), mp 140°. Prepd from HNO_3 and the free base

Refs: 1) Beil, not found 2)P.Grammatikatis, BullSocChimFr **1952**, 446-538 & CA **47**, 3817 (1953) (Infrared spectrum)

1-Ethylideneamino-3-nitroguanidine, $CH_3CH{=}N\,NH\,C(=NH)NHNO_2$; mw 145.12, N48.26%, mp 234° (Ref 2) 144-5° (Ref 4). Prepd by reacting acetaldehyde and nitroaminoguanidine with a little acetic acid

Refs: 1)Beil, not found 2)G.B.L.Smith & E.P.Shoub, JACS **59**, 2078(1937) & CA **32**, 118(1938) 3)E.Lieber et al, AnalChem **23**, 1594-1604 (1951) & CA **46**, 3857(1952)(Infrared spectrum) 4)R.A.Henry & G.B.L.Smith, JACS **74**, 278 (1952) & CA **47**, 8020 (1953)

Ethylidene Aniline and Derivatives
Ethylidene Aniline, $CH_3.CH:NC_6H_5$; mw 119.17, N 11.76%; dark red-brown viscous liq, sp gr?, fr p?, bp 205°; insol in w, sol in gasoline & benz. Can be prepd by the action of acetaldehyde on aniline, followed by distillation. Used as aceelerator in rubber vulcanization
Refs: 1) Beil **12**, 188 2) W.Miller, et al, Ber **25**, 2030-32 (1892) 3) CondChemDict (1961), 472-L

β-Nitroethylidene Aniline, $O_2NCH_2CH:NC_6H_5$; mw 164.16, N 17.07%; ndl (benz) mp 94-5° (sinters at 90°). Prepd from aniline.HCl and acidified methazonic acid
Refs: 1) Beil **12**, 188 & [110] 2) W.Meister, Ber **40**, 3447 (1907)

Ethylidene -2-4-Dinitroaniline, $CH_3CH:NC_6H_3(NO_2)_2$; mw 209.16
N 20.09%, yel ndl(w/alc), mp 196°, sol in 20% HCl & dil alkali. Prepd by boiling with *1N* NaOH the triethylamine salt of HO_2C CH (2,4-dinitroaniline) CH_2(2,4-dinitrophenol) - the amine salt of O,N-bis(2,4-dinitrophenyl)-serine – and then acidifying. The compd may actually be N-vinyl-2,4-dinitroaniline, but the authors made no attempt to find out
Refs: 1) Beil, not found 2) T.Wagner-Jauregg & J.H.Short, Ber **89**, 253-6 (1956) & CA **50**, 14537 (1956)

β-Nitroethylidene-4-Nitroaniline, $O_2NCH_2CH:NC_6H_4(NO_2)$; mw 209.16, N 20.09% ndl (chlf), dec 183°. Prepd from 4-nitroaniline and methazonic acid
Refs: 1) Beil **12**, 717 2) W.Meister, Ber **40**, 3447 (1907)

Ethylidene Benzoacetate. See Ethylidene Acetobonzoate in this Vol

Ethylidene Chloride or Ethylidene Dichloride. See Dichloroethane in Vol 5 of Encycl, p D1209-R

Ethylidene Diacetamide, $CH_3CH(HN.CO.CH_3)_2$; mw 144.17, N 19.43%; col pr, mp 169° (Refs 2 & 6) 180° (Refs 3, 7 & 8), sp gr 1.231. Prepd by heating in a sealed tube acetaldehyde and acetamide (Ref 2); later by refluxing the same with HAc (Ref 3), then using acetonitrite and acetaldehyde (Ref 6), and diethylacetal/acetamide (Ref 7). Attempted nitration at 15° with HNO_3/Ac_2O was a failure (Ref 4)

Its nitration was conducted in A.D.Little laboratory in the following manner (Ref 5): To the stirred soln of 1.44g (0.01 mol) of ethylidine diacetamide in 4ml (0.04mol) of acetic anhydride contd in a 25-ml Erlenmeyer flask (cooled in ice-HCl bath) was added dropwise 1.26g (0.02 mol) of absolute nitric acid. The resulting pale-blue soln was poured over 25g of cracked ice and the resulting soln was neutral by addn of Na carbonate. This soln was yellow in color but no ppt was obtd

When attempted nitration was conducted at temp of ice-water bath, a fume-off resulted after all the amide was added to the nitration soln
Refs: 1) Beil **2**, 179 2) N.Tawildaron, Ber **5**, 477 (1872) 3) W.A.Noyes & D.B. Forman, JACS **55**, 3493 (1933) & CA **27**, 4775 (1933) 4) R.C. Brain & A.H.Lamberton, JCS **1949**, 1633-5 & CA **44**, 1411 (1950) 5) A.D.L, Synthesis HE's 2nd Report (1951), pp 51 & 54 6) Vereinigte Glanzstoff-Fabriken A-G, Brit P710468 (1954) & CA **49**, 11709 (1955) 7) H. Boehme & G. Berg. Ber **99**, (7) 2127-35 (1966) & CA **65**, 8873 (1966) 8) N. Yanaihara & M. Saito, ChemPharmBull (Tokyo) **15**(1), 128 (1967) & CA **66**, 104653 (1967)

Ethylidene Diacetate or Diacetoxyethane, $CH_3.CH(O_2C.CH_3)_2$; mw 146.145, col liq, sp gr 1.061 at 12°, mp 18.85°, bp 168° at 740mm; sl sol in w, miscible with alc, decompd by alkalies. Can be prepd by heating acetaldehyde with acetic anhydride at 180°. Used for making acetic anhydride and vinyl acetate
Refs: 1) Beil **2**, 152, (71) & [167] 2) A. Geuther, Ann **106**, 250 (1858)

Ethylidene Dichloride. See Dichloroethylidene in Vol 5, p D1209-R

Ethylidene Diethylether. See Acetal in Vol 1 of Encycl, p A13-L

Ethylidene Difluoride or 1,1-Difluoroethane, CH_3CHF_2; mw 66.05; col, odorless gas, sp gr 1.004 at -25°, fr p -117°, bp -24.7°, insol in w. Can be prepd by adding hydrogen fluoride to acetylene. Used as aerosol propellant, refrigerant and intermediate

Refs: 1) Beil, not found 2)A.L.Henne & M.W. Renoll, JACS **58**, 887-9 (1936) & CA **30**, 5176 (1936) 3) CondChemDict (1961), 381-R (1,1-Difluoroethane) 4) Sax, 3rd edit (1968), p 666-R

Ethylidene Dimethylether or Dimethylacetal, $CH_3CH(OCH_3)_2$; mw 90.12; col, flammable, liq, sp gr 0.850 at 20/40°, fr p -113.2°, bp 64° at 748mm; miscible with w, alc, eth & chlf. Can be prepd by heating acetaldehyde with methanol & glac AcOH, followed by distillation and rectification. Used in medicine and in organic synthesis

Refs: 1)Beil **1**, 603,(326) & [671] 2) A.Wurtz, AnnCh [3] **48**, 373 (1856) 3)Cond ChemDict (1961), 391-R (Dimethylacetal) 4)Sax, 3rd edit (1968), p 680-L

Ethylidene Diperoxide. Misnomer for Diethylidene Diperoxide described in Vol5 of Encycl, p D1238-L

Ethylidene Diurethane, $CH_3CH(NH.CO_2.C_2H_5)_2$; mw 204.22, N 13.72%; ndls, mp 120-121° (Nencki); mp 125-126° (Lange), bp 170-180° at 20mm, sol in alc, eth and hot w. Was first prepd in 1874 by interaction of acetaldehyde and urethane (Refs 1 & 2)

ADL, Inc (Ref 3) prepd it by shaking in 250-ml flask: 35.6g (0.4mol) of urethane, 8.9g (0.2mol) of acetaldehyde in 200ml distd w contg 0.6ml of concd HCl and allowing to stand for 24 hours. Then the ppt was collected and recrystallized from water

Nitration was attempted by adding drop by drop with stirring, 4.08g (0.02 mol) of ethylidene diurethane into a flask (cooled in ice-HCl bath), contg 2.5g (0.04 mol) of absol nitric acid mixed with 8.0g (0.08 mol) of acetic anhydride. During addition it was necessary to wait until each drop dissolved before the addition of next drop. After adding all 4.08g of ethylidene diurethane, the soln turned blue and at that moment it was poured over cracked ice. As result of this an oily paste settled at the bottom of vessel contg ice. Extraction with ether gave 1.6g of pasty solid product melting at 118.5-119.5°. A mixed mp with Ethylidene Diurethane showed no depression

Note: It is necessary to pour the nitrated liquid over ice, immediately after it becomes blue because delay will cause its decomposition with vigorous evolution of gas and loss of product

Refs: 1) Beil **3**, 24,(11) 2)M.Nencki, Ber **7**, 160 (1874) 3)ADL, Synthesis HE's, 2nd Report (1951), 54-5

4,6-Ethylidene-methylglucosides and Derivatives

Ethylidene -α-methylglucoside

$\overline{OCH(OCH_3)CHOHCHOHCHCHCH_2OCH(CH_3)O}$;

mw 220.22, mp 77°. Prepd by reacting acetylene with α-methylglucoside in EG under 10° in the presence of $HgSO_4$ & H_2SO_4

Refs: 1)Beil, not found 2)H.S.Hill & H.Hibbert JACS **45**, 3108-3116 (1923) & CA **18**, 1987 (1924) 3)B.Helferich & H.Appel, Ber **64B**, 1841-7 (1931) & CA **25**, 5401 (1931)

4,6-Ethylidene-β-methylglucoside, see formula above; mw 220.22, ndls, sp gr 1.40, mp 182-83°; Prepd by hydrolysis with NaOEt in chlf of diacetylethylidene-β-methyl-d-glucoside, which was prepd from β-methylglucoside, Ac_2O & pyridine

Refs: 1)Beil, not found 2)B.Helferich & H. Masamune, Ber **64B**, 1257-60 (1931) & CA **25**, 5147 (1931) 3)H.Appel & W.N.Haworth, JCS **1938**, 793-7 & CA **32**, 6626 (1938)

4,6-Ethylidene-α-2-methylglucoside Nitrate

$\overline{OCH(OCH_3)CH(ONO_2)CHOHCHCHCH_2OCH(CH_3)O}$;

mw 265.22, N 5.28%, ndls(alc+petr eth), mp 133°. Prep by refluxing the α-2,3-dinitrate (see below) in alcoholic hydrazine

Refs: 1)Beil, not found 2)K.S.Ennor & J.Honeyman, JCS **1958**, 2586-94 & CA **52**, 19960 (1958)

4,6-Ethylidene-β-2-methylglucoside Nitrate, see formula for *a*; mw 265.22, cryst, mp 144°. Prepd by refluxing the β-2,3-dinitrate (See below) in alcoholic hydrazine

Refs: 1)Beil, not found 2)K.S.Ennor & J.Honeyman, JCS **1958**, 2586-94 & CA **52**, 19960 (1958)

4,6-Ethylidene -α- 3-methylglucoside Nitrate, $\overline{OCH(OCH_3)CHOHCH.(ONO_2)}\underline{CHCHCH_2OCH(CH_3)O}$ mw 265.22, N 5.28%; mp 172-3°. Prepd by alkaline hydrolysis of the α-2,3-dinitrate (See below)

Refs: 1)Beil, not found 2)E.G.Ansell & J. Honeyman, JCS **1952**, 2778-89 & CA **47**, 1066 (1953)

4,6-Ethylidene-β-3-methylglucoside Nitrate, see formula for *a*; mw 265.22, mp 146-8°. Prepd by heating the β-2,3-dinitrate (see below) for 1hr at 100° in a sealed tube with NaI and acet

Refs: 1)Beil, not found 2)D.J.Bell & R.L.M.Synge, JCS **1938**, 833-6 & CA **32**, 6627 (1938)

4,6-Ethylidene-α-2,3-methylglucoside Dinitrate, $\overline{OCH(OCH_3)CH(ONO_2)CH(ONO_2)}\underline{CHCHCH_2OCH(CH_3)O}$; mw 310.22, N 9.03%; mp 96-7° (Ref 2), 101° (Ref 3). Prepd by addition of the parent in chlf to N_2O_5 in chlf at 0°. Later prepd by adding to the parent in Ac_2O at 0°, fuming HNO_3 in Ac_2O (Ref 3)

Refs: 1)Beil, not found 2)E.G.Ansell & J.Honeyman, JCS **1952**, 2778-89 & CA **47**, 1066 (1953) 3)J. Honeyman & J.W.W.Morgan, JCS **1955**, 3660-74 & CA **50**, 9298 (1956)

4,6-Ethylidene-β-2,3-methylglucoside Dinitrate, see formula for *a*; mw 310.22, N 9.03%; ndls (pet eth or CH_3OH), mp 88-9°. Prepd by addition of the parent in chlf to N_2O_5 in chlf at 0°. Later prepd by adding fuming HNO_3 in Ac_2O to the parent in Ac_2O at 0° (Ref 3)

Refs: 1)Beil, not found 2)D.J.Bell & R.L.M.Synge, JCS **1937**, 1711-18 & CA **32**, 504(1938) 3)J. Honeyman & J.W.W.Morgan, JCS **1955**, 3660-74 & CA **50**, 9298(1956)

Ethylideneperoxide (Äthylidenperoxyd- in Ger) (Was called by Baeyer and Villiger Acetaldehydperoxyd or Diacetaldehyd-diperoxyd and by Wieland and Wingler, Diäthyliden-diperoxyd),

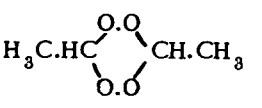
bp-expl violently when heated in a closed tube; does not seem to be sensitive to friction; insol in water, easily sol in eth, more difficultly in alc, still more difficultly in benz and very diff in petroleum ether. When left standing for several hours in a closed container, the oily substance changes into a *tough tar*, extremely sensitive to impact and friction

The following discussion is mainly historical and indicates the confusion generated by non-analyzable materials

Two modifications were prepd and closely examined before WWII by Rieche and Meister (Ref 4). Among the several methods of preparation, they recommended the one from an ethereal solution of α-hydroxyethylhydroperoxide (α-Oxyäthylhydroperoxyd-in Ger) and P_2O_5

A compound which closely resembled the tarry substance was prepd in 1900 by Baeyer and Villiger by adding 0.1g of acetaldehyde to a mixture of 1g H_2O_2 (10% sol) and 2g SO_3, cooled to 0°. The oil which separated on standing gave, on cooling in ice, a volatile solid which exploded violently on impact or heating. They called the compound *Diacetaldehydperoxyd*, but did not determine its composition

A substance closely resembling the oil produced by Rieche and Meister was prepd previous to them by Wieland and Wingler (Ref 3) by treating freshly distilled acetaldehyde with an ethereal solution of anhydrous H_2O_2. It exploded on heating in a closed container. W&W. called it *Diäthyliden-diperoxyd* and assigned the formula



It might also be called Diacetaldehydediperoxide or Acetaldehydeperoxide.

The chemical properties of ethylideneperoxide are given in Ref 4

Rohrlich and Sauermilch (Ref 5), in an article on Tricycloacetoneperoxide, said that they determined the lead block expansion for a 10g sample of the "Azetaldehyd" of Baeyer and Villiger. The value was 330cc as against 250cc for Tricycloacetoneperoxide. They also stated that when a test tube with a sample of Acetaldehydeperoxide was dropped on the floor from a height of 2m, a violent detonation took place. The same authors stated that they could not determine the velocity of deton of acetaldehydeperoxide because it was very dangerous to handle. (Compare with Diethylene Diperoxide, described in Vol 5 of Encycl, p D1238-L)

Refs: 1)Beil **19** [449] 2)A.Baeyer & V. Villiger, Ber **33**, 2484 (1900) 3)H.Wieland & A. Wingler, Ann **431**, 315 (1923) 4) A. Rieche & R. Meister, Ber **64**, 2335 (1931) & **65**, 1274 (1932) 5)M.Rohrlich & A. Sauermilch, SS **38**, 98 (1943) 6)Tobolsky & Mesrobian (1954), not found 7)J.d'Ans et al, AngewChem **66**, 633-35 (1954) & CA **49**, 10832 (1955)

Ethylidene Peroxide, Dimeric, $CH_3CHOOCH(CH_3)OO$; mw 120.11, ndls, mp 63°, extremely vol; sol in organics; insol w. Prepd by heating at 60-70° and 14mm Hg dimeric triethylene ozonide (Ref 4 above). C-H analysis gave correct empirical formula while molecular weight determination gave 117 in benz and 123-7 in dioxane. Stable at 100°, doesn't liberate much [0] during iodometric titration. Authors of Ref 2 believe that no "monomer" exists only the dimer and polymers, and consider the "dimer" of Ref 3 above to be the polymer. Absolutely minimal quantities must be prepd because of its unpredictable detonability

Refs: 1)Beil **19**, [449] 2)A.Rieche & R. Meister, Ber **72B**, 1933 (1939) & CA **34**, 712 (1940) 3)Tobolsky & Mesrobian (1954), 179

Ethylideneperoxide, Polymeric, $(CH_3CHOO-)_x$; mw(60.05)$_x$ viscous oil, sp gr 1.160-1.165, n_D 1.451 at 19°. Prepd by treating $CH_3CH(OH)OOH$ or $[CH_3CH(OH)O]_2$ with P_2O_5, using heat with the latter ether. The freshly prepd material is fairly safe to handle, but, rapidly in air, and in a few days in a closed container, becomes highly sensitive to contact with a glass rod (but, strangely, not to the shock of the detonation caused thereby-only the contacted material goes off)

Refs: 1)Beil **4**, [449] 2)A.Rieche & R.Meister, Ber **64**, 2328 & 2335 (1931) 3)Ibid, AngewChemie **49**, 101(1936) 4)Ibid, Ber **72B**, 1933(1939) & CA **34**, 712(1940) 5) Tobolsky & Mesrobian (1954), 179 6)J.Malicki, PrzemyslChem **32**, 7-9(1953) & CA**48**, 4428(1954)

Ethyliodoacetate. Brit tear gas, designated **SK**, listed in Vol 2 of Encycl, p C169-R, under CHEMICAL AGENTS. See Also Cond ChemDict (1961) 472-R

Ethyl Lactate and Derivatives

Ethyl Lactate, $CH_3.CHOH.COOC_2H_5$; mw 118.13; col, oily liq, with mild odor; sp gr 1.030 at 25/4°, bp 154-155°, fl p (Tag open cup) 158°F; miscible with w, alc, eth, esters, ketones, hydrocarbons & oils. Was prepd from Etl and the silver salt or by heating the acid with alc at 170° in a closed tube. Two methods of prepn are listed in Ref 3: a)By esterification of lactic acid with ethanol and b)By combining acetaldehyde with hydrocyanic acid to form acetaldehyde cyanohydrin, and this is treated with ethanol & HCl to ethyl lactate. Used as a solvent for cellulose acetate and nitrate, other cellulose esters, resins, lacquers, paints and enamels

Refs: 1)Beil **3**, 264, 267, 280,(102,109) & [185, 187, 205] 2)Klimenko, J Russ Phys-ChemSoc **12**, 25(1880) 3)CondChemDict (1961), 473-L

Ethyl Lactate Nitrate, $CH_3CH(ONO_2)CO_2C_2H_5$; mw 163.13, N8.59%; liq, sp gr 1.1824 at 20°/4, bp 73-4°(10mmHg), 178°, n_D 1.4155 at 20°. Prepd from the parent and mixed nitrating acid

(Ref 2), later from the 2-bromo ester and silver nitrate in acetonitrile (Ref 3), by oxidizing 1-nitropopane-1 with nitric acid followed by esterification (Ref 4), by reacting silver nitroform with the 2-bromoester (Ref 5) and by reacting the 2-bromo or iodoester with nitric acid (Ref 6)

Ref: 1)Beil **3**, 282 2)L.Henry, Ber **3**, 532 (1870) 3)A.F.Ferris, et al, JACS **75**, 4078 (1953) & CA **49**, 8158 (1955) 4)V.M.Belikov et al, ZhObshcheiKhim **30**, 191-2 (1960) & CA **54**, 22350 (1960) 5)G.S.Hammond, et al, Tetrahedron **19**, Suppl 1, 177-95 (1963) & CA **59**, 11237 (1963) 6)N.V.Svetlakov et al, ZhOrgKhim **4**(12)(1968), 2096-9 & CA **70**, 67510 (1969)

Ethyl-2-Nitrolactate Nitrate
$CH_3C(NO_2)(ONO_2)CO_2C_2H_5$; mw 208.13, N13.46%, OB to CO_2-53.4%; liq, bp 80° (0.1mm Hg), n_D L4432. Prepd by addition of $HO_2CCH(CH_3)CO_2C_2H_5$ to 100% HNO_3 at 0°, then letting stand one day at ambient temp

Refs: 1)Beil, not found 2)L.W.Kissinger & H.E.Ungnade, JOC **23**, 1340-2 (1958) & CA **53**, 15951 (1959)

Ethyl-3-Nitrolactate, $CH_2(NO_2)CNOHCO_2C_2H_5$; mw 163.13, N8.59%, liq, bp 100°(4mm Hg), n_D 1.4520 at 20°. Prepd by adding to $CHOCO_2C_2H_5$ the sodium salt of nitromethane in methanol, under 5°

Refs: 1)Beil, not found 2)L.A.Yanovskaya, etal, IzvAkadNaukSSSR, SerKhim **1964** (11), 2093-5 & CA **62**, 7630 (1965)

Ethyl-3,3-Dinitrolactate, $CH(NO_2)_2CHOHCO_2C_2H_5$; mw 208.13, N13.46%, OB to CO_2-53.4% liq, n_D 1.4573 at 25°. Prepd by adding the half (ethyl)acetal of ethylglyoxalate to $KCH(NO_2)_2$, and acidifying the intermediate **potassium salt** (mp 144° dec)

Refs: 1)Beil, not found 2)L.W.Kissinger et al, JOC **22**, 1658-62 (1957) & CA **52**, 8963 (1958)

Ethyl Malonate or Diethyl Malonate, $CH_2(CO_2C_2H_5)_2$; mw 160.17, col liq with sweet ether odor, sp gr 1.055 at 25-25°, fr p −50 to 51.5°; bp 198-199.3°; practically insol in w; sol in alc, eth, chlf & benz. Can be prepd by passing HCl into cyanoacetic acid dissolved in absol ethanol, with subsequent distillation. Used as intermediate for barbiturates and certain pigments

Refs: 1)Beil **2**, 573,(247) & [524] 2) B.Finkelstein, Ann **133**, 349 (1865) 3) CondChemDict (1961), 473-L 4)Sax, 3rd edit (1968), 663-L

Ethylmalonic Acid and Derivatives
Ethylmalonic Acid, $C_2H_5.CH(COOH)_2$; mw 132.11; col prisms, mp 111.5°, bp-decomp at 160°; very sol in w, alc or eth. Can be obtd from α-bromobutyric acid heated with K mercuric cyanide, followed by KOH

Refs: 1)Beil **2**, 643,(275) & [569] 2) J.Wislicenus, Ber **2**, 721(1869) 3)CondChemDict (1961), 473-R

Ethylmalonic Acid Monoazide
$C_2H_5CH(CO_2H)CON_3$, mw 157.13, N26.74%; yel oil, sol in w, eth, chlf & alc; expl weakly on heating. Prepd by diazotization of the K salt of the *monohydrazide*

Refs: 1)Beil **2**,[570] 2)T.Curtius & W. Sieber, Ber **55**, 1555 (1922) & CA **16**, 3883 (1922)

1-Ethyl-*iso*-melamine, $N(C_2H_5)C(=NH)NHC(=NH)NHC(=NH)$; mw 154.175, N54.50%. Prepd by mixing HCl, $C_2H_5NH_2$.HCl, add monopotassium dicyanoguanidide, and heating. Isolated as the hydrochloride salt, dec 312°

Refs: 1)Beil, not found 2)D.W.Kaiser & D.E.Nagy, USP2481758 (1949) & CA **44**, 5926 (1950)

Ethylmercaptan, Ethylsulfhydrate or Ethanthiol, C_2H_5SH; mw 62.13; col, volatile, very unpleasantly garlic smelling liquid; flammable, sp gr 0.83907 at 20/4°, fr p -121°, bp 36-37°. n_D 1.4305 at 20°, fl p < 0°; sl sol in w; sol in alc, eth, petr naphtha & alkalies. Can be prepd by saturating KOH soln with H_2S, mixing with Ca ethylsulfate soln and distilling on a water bath (Ref 4) (See also Refs 1&2). Was proposed for use in self-igniting rocket fuels (Ref 3)

This compd was listed in Vol 1 of Encycl,

p 101-R, among "Acyclic Mercaptans Containing 1-5 Carbon Atoms"
Refs: 1)Beil 1, 340,(171) & [341] 2)A.Kekulé,Ann **90**, 311 (1854) 3)P.C.Condit & M.A.Pino, USP 2750732 (1956) & CA **50**, 16110-12 (1956) 4)CondChemDict (1961), 473-R 5)Sax, 3rd edit (1968), 757-L

Ethyl Methacrylate or Ethyl Methylacrylate, $H_2C:(CH_3).COOC_2H_5$; mw 114.14 col liq, sp gr 0.913 at 15.6°, fr p < -75°, bp 118-119°, fl p (open cup) 95°F; insol in w; sol in alc or eth; readily polymerizes. Can be prepd by reaction of methacrylic acid with ethanol. Was used in prepn of acrylate resins and of other copolymers and polymers. Acrylate resins are thermoplastic polymers or polymers of acrylic and methacrylic acid, esters of these acids or acrylonitrile. Acrylic acid & acrylonitrile and their derivatives are described in Vol 1 of Encycl, pp A96-R & A97. Methacrylic acid is α-methylacrylic acid, $CH_2:C(CH_3)COOH$ described in CondChem Dict (1961), p 723-R

These monomers polymerize readily in presence of light, heat or catalysts (such as benzoyl peroxide) and must always be stored or shipped with inhibitor present to avoid spontaneous and explosive polymerization. The acrylic and methacrylic resins range from soft, sticky, semifluid materials to hard solids. The polymethacrylics are harder than corresponding polyacrylates and the methyl esters form harder resins than the ethyl or butyl esters. Polymethylmethacrylate, examples of which are *Lucite* and *Plexiglas*, is outstanding for its clarity and transparency. An almost unique property is that of carrying light around corners and reflecting it out the edges of a piece of cast polymer. Additional characteristics of acrylate resins are: low water absorption, low sp gr, good shock resistance. They are compatible with many plasticizers and are soluble in aromatic hydrocarbons, chlorinated hydrocarbons, esters and ketones

Their uses are very extensive, such as: aircraft canopies & windows, instrument dials, safety glass, contact lenses, lighting fixtures, refrigerator parts, dentures, paints & finishes, adhesives etc. The synthetic fibers *Orlon*, *Acrylan*, *Dynel* and *Vinyon N* are also acrylate resins or acrylics since they are copolymers of acrylonitrile. Acrylic rubbers, including acrylonitrile rubber are also of this general class
Refs for Ethyl Methacrylate:
1)Beil **2**, 423, (191) & [399] 2)K.Auwers & E.Kobner, Ber **24**, 1935 (1891) 3)E.H.Riddle, "Monomeric Acrylic Esters", Reinhold, N Y(1950) 4)CondChemDict (1961), 474-L 5)Sax, 3rd edit (1968),758-L
Refs:for Acrylate Resins:
A)Kirk & Othmer 1, (1947), 180-84 B)Cond ChemDict (1961), 19 C)Kirk & Othmer 1, (1963), 306-11 D)CondChemDict (1971), 14-R

Ethylmethane Sulfonamide and Derivatives
Ethylmethane Sulfonamide,
$H_3C.SO_2.NH.CH_2.CH_3$; mw 123.175, col liq, sp gr 1.191 at 24°, bp 105.5°(0.3mm Hg), n_D 1.4900 at 25°, sol in w. Prepd by mixing at 0° 1 mole of $C_2H_5NH_3Cl$, 1 mole of CH_3-SO_2Cl, & 2 moles of NaOH in w
Refs: 1)Beil, not found 2)B.Helferich & H.Grünert, Ber **73B**, 1131-3 (1940) & CA **35**, 1027 (1941) 3)C.C.Bard & J.A. Larkin, Belg P 652532 (1964) & CA **64**, 9139 (1966)

Trinitroethyl-methanesulfonamide, $(O_2N)_3C.CH_2NH.SO_2.CH_3$; mw 258.17, N16.27% ($NO_2$) OB to CO_2-18.6%; crysts, sp gr 1.74, mp 155°, ignition temp 258°. Can be prepd by heating methane sulfonamide with the theoretical quantity of Trinitroethanol at 70°(Ref 3), or by allowing the above mixture to stand for several days in methanol soln (Ref 2). Its impact sensitivity approaches that of Pentolite
Refs: 1)Beil, not found 2)Hercules PowderCo, Progress Rept, Oct 1949,High Expls,Contract NOrd 9925 3)W.F.Sager & D.V.Sickman, NAVORD Rept **483**, (1952), Res & Devel in New Chemical High Expls, p 7

N-Nitro-trinitroethyl-methanesulfonamide, $(O_2N)_3C.CH_2N(NO_2).SO_2.CH_3$; mw 303.17, N18.48%($NO_2$) OB to CO_2-2.6%, crysts, sp gr 1.82, mp 52°. Prepd by the nitration of n-Trinitroethylmethane sulfonamide

Its sensitivity to impact is comparable to Pentolite

Refs: 1)Beil, not found 2)R.H.Saunders et al, HerculesPowderCo, Progress Rept Dec 1949 on Contract NOrd 9925 3)W.F.Sager & D.V.Sickman, NAVORD Rept **483**, (1952) Res & Devel in New ChemHighExpls, p 31

Ethylmethyl Aniline and Derivatives

N-Ethyl-N-Methyl Aniline, $C_6H_5N(CH_3)(C_2H_5)$; mw 135.21, N10.36%; liq, sp gr 0.9193 at 55°/4, bp 203-5°, sol in alc & eth. Prepd from ethylaniline & CH_3I. Forms a **picrate**, green-yel prisms, mp 134-5°
Refs: 1)Beil **12**, 162, (156) & [91] 2) A.W.Hofmann, Ann **74**, 152 (1850)

2-Nitro-N-Ethyl-N-Methyl Aniline, $O_2NC_6H_4N(CH_3)(C_2H_5)$; mw 180.21, N15.55%. Prepd by heating diethylsulfate with 2-nitro-N,N-dimethylaniline, and isolation as the **picrate**, mp 126-7°
Refs: 1)Beil, not found 2)H.A. Fahim & M.Galaby, JCS **1950**, 3529-32 & CA **45**, 7037 (1951)

4-Nitro-N-Ethyl-N-Methyl Aniline, $O_2NCH_6H_4N(CH_3)(C_2H_5)$; mw 180.21, N15.55% crysts, mp 88°. Prepd by heating diethylsulfate with 2-nitro-N,N-dimethylaniline to give the quaternary anilinium ethyl sulfate salt which was then heated to give the N-Ethyl-N-Methyl compound
Refs: 1)Beilk not found 2)H.A.Fahim & M. Galaby, JCS **1950**, 3529-32 & CA **45**, 7037 (1951)

N-(2-Nitroethyl)-N-Methyl Aniline, $C_6H_5N(CH_3)(CH_2CH_2NO_2)$; mw 180.21, N15.55%; liq, bp 110° (0.2mmHg), 112° (0.05mmHg) mp-45°, n_D 1.5635 at 20°. Prepd from N-methyl aniline and $CH_2{=}CHNO_2$. The **HCl salt**, mp 82°, is stable
Refs: 1)Beil, not found 2)R.L.Heath & J.D. Rose, JCS **1947**, 1486-9 & CA **42**, 4911 (1948) 3) H. Hopff & M. Capaul, HelvChimActa **43**, 1898-1910 (1960) & CA **55**, 12282 (1961)

2,4-Dinitro-N-Ethyl-N-Methyl Aniline $(O_2N)_2C_6H_3N(CH_3)(C_2H_5)$; mw 225.21, N18.66%; or crysts, sp gr 1.429 at 10° mp 59°(55°). Prepd from 2,4-dinitrochlorobenzene and ethylmethylamine
Refs: 1)Beil **12**, 750 2)J.Graymore, JCS **1938**, 1311-13 & CA **33**, 133 (1939) 3) E.W.Day, Jr., et al, AnalChem 38(8), 1053-7 (1966) (Detection) & CA **65**, 11324(1966)

Ethylmethylbenzenes and Derivatives

4-Ethyl-1-Methyl-benzene, $CH_3C_6H_4C_2H_5$; mw 120.20, oil, sp gr 0.8588 at 20°/4°, bp 162°, n_D 1.4929 at 24.4°. Prepd from 4-bromotoluene, ethyl bromide and sodium in eth
Refs: 1)Beil **5**, 397, (193) & [310] 2)E.Glinzer & R.Fittig, Ann **136**, 312 (1865)

3-Ethyl-1-methyl-benzene, $CH_3C_6H_4C_2H_5$; mw 120.20, liq, sp gr 0.869 at 20°, bp 150°, n_D 1.4965 at 19.8°. Prepd from 3.bromotoluene, ethyl bromide and sodium in eth
Refs: 1)Beil **5**, 396, (192) & [309] 2)E.Wroblewski, Ann **192**, 198(1878)

2-Nitro-4-ethyl-1-methyl-benzene, $CH_3C_6H_3C_2H_5(NO_2)$; mw 165.20, N8.48%; liq, bp 247-9°, 73-6°(1 mm Hg). Prepd by nitration of the parent compd
Refs: 1)Beil, not found 2)O.L.Brady & J.N.E. Day, JCS **1934**, 114-21 & CA **28**, 1993 (1934) 3) M. Dolinsky, et al, AssocOfficAgrChemJ **42**, 709-20 (1959) & CA **54**, 4268 (1960) (Prepn & infrared spectrum)

3-Nitro-4-ethyl-1-methyl-benzene, $CH_3C_6H_3C_2H_5(NO_2)$; mw 165.20, N8.48%; liq, bp 115-20° (10mm Hg), 69-73° (1 mm Hg). Prepd by hydrazine reduction of 3-nitro-4-ethylbenzaldehyde (bp 155° at 10 mm), from 4-ethylbenzaldehyde and KNO_3 in H_2SO_4 at 0°
Refs: 1)Beil, not found 2)I.J.Rinkes, RecTravChim **64**, 205-13(1945) & CA **40**, 4034 (1946) 3)M.Dolinsky etal, AssocOfficAgrChemJ **42**, 709-20 (1959 & CA **54**, 4268 (1960) (Prepn & infrared spectrum)

2-Nitro-3-ethyl-1-methyl-benzene, $CH_3C_6H_3C_2H_5(NO_2)$; mw 165.20, N8.48%; liq bp 72-6° (1 mm Hg). Prepd from the parent and a mixture of concd sulfuric and 70% nitric acids at 0-5°
Refs: 1)Beil, not found 2)M.Dolinsky et al, AssocOfficAgrChemJ **42**, 709-20 (1959) & CA **54**, 4268 (1960)(Also infrared spectrum)

4-Nitro-3-ethyl-1-methyl-benzene,
$CH_3C_6H_3C_2H_5(NO_2)$; mw 165.20, N8.48%; liq, bp 253-63°, 70-1°(0.7 mm Hg). Prepd from 4-ethyl-2-methyl-acetanilide by HNO_3/H_2SO_4- mp 143°, hydrolysis to the aniline-mp 74°, and diazotization/removal of the amine group
Refs: 1)Beil, not found 2)G.T.Morgan & A.E.J.Pette, JCS **1934**, 418-22 & CA **28**, 4396-7 (1934) 3)M. Dolinsky et al, Assoc-OfficAgrChem J **42**, 709-20 (1959) & CA **54**, 4268 (1960) (Prepn & infrared spectrum)

5-Nitro-3-ethyl-1-methyl-benzene
$CH_3C_6H_3C_2H_5(NO_2)$; mw 165.20, N8.48%; liq, bp 263-4°. Prepd from 4-ethyl-2-methyl-acetanilide by HNO_3/HAc-mp 142°, hydrolysis to the aniline - mp 64°, and diazotization/removal of the amine group
Refs: 1)Beil, not found 2)G.T.Morgan & A.E.J.Pette, JCS **1934**, 418-22 & CA **28**, 4396-7 (1934)

6-Nitro-3-ethyl-1-methyl-benzene,
$CH_3C_6H_3C_2H_5(NO_2)$; mw 165.20, N8.48%, liq, bp 245°, 70-1°(0.5 mm Hg). Prepd from the parent with HNO_3/HAc in the cold
Refs: 1)Beil **5**, [310] 2)A.Mailhe, CR **173**, 160-2 (1921) & CA **15**, 3985 (1921) 3) M.Dolinsky et al, AssocOfficAgrChem J **42**, 709-20 (1959) & CA **54**, 4268 (1960) (Prepn & infrared spectrum)

2,3-Dinitro-4-ethyl-1-methyl-benzene,
$CH_3C_6H_2C_2H_5(NO_2)_2$; mw 210.20, N 13.33%; plates (alc), mp 51-2°, sol in boiling alc. Prepd from the parent with cold fuming nitric acid
Refs: 1)Beil **5**, 399 2)P.Jannasch & A. Dieckmann, Ber **7**, 1514 (1874) 3)O.L. Brady & J.N.E.Day, JCS **1934**, 114-2 & CA **28**, 1993 (1934)

2,6-Dinitro-4-ethyl-1-methyl-benzene,
$CH_3C_6H_2C_2H_5(NO_2)_2$; mw 210.20, N13.33%; mp 62.5°. Prepd from the 2,3,6-trinitro derivative (see below) by reaction with ammonia to give the 3-amino, mp 143°, which was reacted in alc with HNO_2 to remove the amino group
Refs: 1)Beil, not found 2)O.L.Brady & J.N.E.Day, JCS **1934**, 114-21 & CA **28**, 1993 (1934) 3)I.J.Rinkes, RecTravChim **64**, 205-13 (1945) & CA **40**, 4034 (1946)

3,5-Dinitro-4-ethyl-1-methyl-benzene,
$CH_3C_6H_2C_2H_5(NO_2)_2$; mw 210.20, N13.33%; yel crysts, mp 48.5°. Prepd from 5-ethyl-2-methyl-acetanilide by nitration at -10°- mp 177-8°, hydrolysis to the aniline-mp 186°, followed by diazotization/removal of the amino group
Refs: 1)Beil, not found 2)I.J.Rinkes, RecTravChim **64**, 205-13 (1945) & CA **40**, 4034, (1946)

Dinitro-3-ethyl-1-methyl-benzene,
$CH_3C_6H_2C_2H_5(NO_2)_2$; mw 210.20, N13.33%; oil, steam distils. Prepd from the parent compd and cold fuming nitric acid. Not further identified
Refs: 1)Beil **5**, 397 2)E.Bartow & A.W.Sellards, JACS **27**, 372 (1905)

2,3,5-Trinitro-4-ethyl-1-methyl-benzene
$CH_3C_6HC_2H_5(NO_2)_3$; mw 255.20, N16.47%; mp 82°. Prepd from the 3,5-dinitro compound and $HNO_3/H_2SO_4/SO_3$
Refs: 1)Beil, not found 2)I.J.Rinkes, Rec TravChim **64**, 205-13 (1945) & CA **40**, 4034 (1946)

2,3,6-Trinitro-4-ethyl-1-methyl-benzene,
$CH_3C_6HC_2H_5(NO_2)_3$; mw 255.20, N16.47%; col prisms (alc), mp 93°, sl sol in cold alc. Prepd from the parent compd and HNO_3/H_2SO_4 by warming
Refs: 1)Beil **5**, 399 2)E.Glinzer & R.Fittig, Ann **136**, 314 (1865) 3)O.L.Brady & J.N.E.Day, JCS **1934**, 114-21 & CA **28**, 1993 (1934) (Prepn) 4)I.J.Rinkes, RecTrav Chim **64**, 205-13 (1945) & CA **40**, 4034 (1946) (Prepn)

2,4,6-Trinitro-3-ethyl-1-methyl-benzene,
$CH_3C_6HC_2H_5(NO_2)_3$; mw 255.20, N16.47%; plates (alc) mp 86°, 90°. Prepd from the dinitro compound by fuming nitric/concd sulfuric acids
Refs: 1)Beil **5**, 397 & [310] 2)E. Bartow & A.W.Sellards, JACS **27**, 372 (1905) 3) A.Mailhe, CR **173**, 160-2 (1921) & CA **15**, 3985 (1921)

Ethylmethylbiimidazole and Derivatives, *4-Ethyl-4′-methyl-2,2′-biimidazole,* $\overline{NH.CH=C(CH_3)N=C}-\overline{C=NC(C_2H_5)=CHNH}$; mw 176.223. This compound is not mentioned in the open literature

Refs: 1)Beil, not found 2)CA, not found

4-Ethyl-4′-methyl-1,1′,5,5′-tetranitro-2,2′-biimidazole, $\overline{N(NO_2)C(NO_2)=C(CH_3)N=C}-\overline{C=NC(C_2H_5)=C(NO_2)N(NO_2)}$; mw 356.21, N15.73% ($NO_2$), OB to CO_2-62.9%, mp 226° (dec). Prepd by adding diethylsulfate to the sodium salt of the tetranitrobiimidazole to make the 4-ethyl compound (mp 250° dec as the dihydrate), and then reacting with diazomethane to give 4′-methyl

Refs: 1)Beil, not found 2)K.Lehmstedt, Ann **507**, 213-25(1933)& CA **28**, 767 (1934)

Ethylmethylcarbamate. See Ethylmethylurethane

Ethylmethyl Carbinol. See Butan-1-ol under Butanol in Vol 2, p B372-R

N-Ethyl-methylenediamine. See Diaminopropane in Vol 5 of Encycl, p D1142

Ethylmethyl Ether or Methylethyl Ether, $C_2H_5.O.CH_3$; mw 60.09; col, volatile, flammable, liq; sp gr 0.697 at 21.1/4°(Lange), 0.725 at 0/0°, bp 7.6°(Lange), 10.8°(Ref 3), n_D 1.3420 at 4°; sol in w, acet miscible with alc & eth. Can be prepd by heating CH_3I with alc or C_2H_5I with CH_3OH. Used as a solvent

Refs: 1)Beil **1**, 314,(158) & [311] 2) E.Busse & K.Kraut,Ann **177**, 272 (1875) 3)CondChemDict (1961), 474-L 4)Sax, 3rd edit (1968), p 758-L

Ethylmethylethyl Ether and Derivatives *Ethyl-α-methylethyl Ether* or *Ethyl Isopropyl Ether,* $CH_3CH(CH_3)OC_2H_5$; mw 88.15, liq, sp gr 0.720 at 25/4°, bp 63-4°, n_D 1.3698 at 25°; sol in w, acet, chlf, alc & eth. Prepd by heating to 150° isopropyl iodide, triethylamine and ethanol

Refs: 1) Beil **1**, 362 & [381] 2)E.Reburel, JahresbFortschrChem **1881**, 409

2-Nitroethyl Isopropyl Ether, $CH_3CH(CH_3)OCH_2CH_2NO_2$; mw 135.15, n_D 10.52%; liq, bp 43°(0.5 mm Hg). Prepd by refluxing 1,2-dinitroethane with isopropyl alcohol

Refs: 1)Beil, not found 2)A.Lambert,et al, JCS **1947**, 1474-7 & CA **42**, 4907 (1948)

Ethyl-α-Nitromethylethyl Ether, $CH_3CH(CH_2NO_2)OC_2H_5$, mw 135.15, N 10.52%; liq, sp gr 1.0314 at 20°, bp 69°(11 mm Hg), 42°(1 mm Hg), n_D 1.4200 at 20°. Prepd by refluxing alc with 1-nitro-2-nitropropane

Refs: 1)Beil, not found 2)C.W.Scaife, Brit P604,360 (1948) & CA **43**,665 (1949) 3)V. I. Burmistrov et al, IzvVysshUcheb-Zaved, KhimKhimTekhnol **1968**, **11**(11), 1257-60 & CA **70**, 77234 (1969)(Prepn)

Ethyl-α-Methyltrinitroethyl Ether, $C(NO_2)_3CH(CH_3)OC_2H_5$; mw 225.15, N18.66%, OB to CO_2-53.3%; liq, sp gr 1.3145 at 20/20°, bp 71-3°(3 mm Hg), 63-4.5°(0.6mm Hg), n_D 1,4386 at 20°. Prepd by reacting nitroform with ethyl vinyl ether in dioxane

Refs: 1)Beil, not found 2)H.Shechter & H.L.Cates,Jr JOC **26**, 51-3(1961) & CA **55**, 19758 (1961) 3)V.I. Grigos & L.T.Eremenko, IzvAkadNauk SSSR, Ser Khim **1969**,(11), 2566-8 & CA **72**, 66293(1970)

Ethylmethylisoxazole and Derivatives *3-Ethyl-5-methyl-isoxazole,* $\overline{N=C(C_2H_5)CH=C(CH_3)O}$; mw 111.14, N12.60%; liq, bp 90°(73 mm Hg), n_D 1.4450 at 20°. Prepd by reacting at 0° 1-propynyl magnesium bromide with propionohydroxamic chloride in eth. Other preps are given in the same Ref, but the 5-ethyl-3-methyl isomer was also formed and it was not possible to separate the two

Refs: 1)Beil, not found 2)H.Feuer & S.Markofsky, JOC 29 (4), 938 (1964) & CA **60**, 13234 (1964)

Note: See under Eulite in this Vol for a trinitro derivative of this isoxazole

Ethylmethyl Ketone or Methylethyl Ketone. See Butanone in Vol 2 of Encycl, p B374-R

Ethylmethyl Ketone Peroxide (Dimolekulares Methyläthylketonsuperoxyd, in Ger), $C(CH_3)(C_2H_5)OOC(CH_3)(C_2H_5)OO$; mw 176.215 liq, sp gr 1.042 at 15°, insol in w, sol alc, eth & benz. Prepd from ethylmethylketone and 2½ % H_2O_2 in the presence of H_2SO_4. Distillable only in the presence of w, 48°, (80 mm Hg), 56° (130 mm Hg); expl on heating over 100° or by stirring with concd H_2SO_4. Sold commercially as a 30-60% soln of a mixture of compounds corresponding to breaking one or both -0-0-linkages (hydroperoxides, hydroxy-hydroperoxides, etc.); used to initiate polyester resin polymerization

Lupersol DDM is the trade name of Wallace & Tieman, Inc, Lucidol Div, 1740 Military Rd, Buffalo, NY, 14205

Refs: 1)Beil **1**,668 2) M. Pastureau, CR **144**, 91(1907) 3)CondChemDict (1961), pp 474-L, 683-L & 739-R 4)K&O, Vol **14**, (1967), p813 5)Sax, 3rd edit (1968), p 758-R 6)K&O, Vol **20**, (1969), pp 825-7

Ethylmethylketone-semicarbazone and Derivatives

Ethylmethylketone-semicarbazone, C_3H_8:C:N.NH.CO.NH_2; mw 129.16, N32.54%; leaflets (from w), sp gr?, mp 143-148°, 135-136°, soly in w 10%; miscible with alc & eth. Can be prepd from ethyl methyl ketone and semicarbazide hydrochloride in the presence of potassium acetate

Refs: 1)Beil **3**, 102, (48) & [82] 2)M. Scholtz, Ber **29**, 610 (1896)

Ethylmethylketone-semicarbazone Azide or α-Azido-ethylmethylketone-semicarbazone, $CH_3CH(N_3)C(CH_3)$=NNHCONH$_2$; mw 170.17, N49.38%; plates (benz or pet eth), mp 94°, sl sol in w, sol in usual organics. Prepd from α-azidoethyl methyl ketone and semicarbazide hydrochloride in the presence of sodium acetate and w

Refs: 1)Beil **3**, 102 2) M.O.Forster & H.E.Fierz, JCS **93**, 676 (1908)

Ethylmethylketone-semicarbazone Azide or Ethyl Azidomethylketone-semicarbazone, $C_2H_5C(N_3)$=NNHCONH$_2$; mw 170.17, N49.38%; ndls (benz) mp 101°, sol in hot w, Prepd from ethyl azidomethyl ketone and semicarbazide hydrochloride in the presence of sodium acetate and water

Refs: 1)Beil **3**, 102 2)M.O.Forster & H.E. Fierz, JCS **93**, 677 (1908)

Ethylmethyl Nitrone and Derivatives. See under 3-Aza-4-oxa-hexene-2 in Vol 1 of Encycl, p 517-L for the parent and a trinitro derivative, the latter originally thought possibly to be a nitrone, but later proved otherwise.

Ethylmethyl Peroxide, $C_2H_5.O.O.CH_3$; mw 76.09; col liq; sp gr 0.8337 at 17/4°, fr p -68 to -69.5°, bp 40° at 740mm, N_D1.3590 at 17°, diff sol in w; easily sol in alc, eth, hexane and many other org solvents. Can be prepd by treating ethylhydroperoxide (qv) with dimethylsulfate in 50% KOH soln in atmosphere of nitrogen, first in the cold and then at 70°

It expl on heating or impact

Refs: 1)Beil **1**, [325] 2)A.Rieche, Ber **62**, 224 (1929) 3)Tobolsky & Mesrobian (1954), 164

Ethylmethylphenyl Ether and Derivatives

Ethyl-3-methylphenyl Ether $C_6H_4(CH_3)OC_2H_5$; mw 136.18, liq, sp gr 0.9650 at 0/0°, bp 192° Prepd from m-cresol, ethyl iodide and KOH

Refs: 1)Beil **6**, 376, (186) & [352] 2) A.Oppenheim & S.Pfaff, Ber **8**, 887 (1875)

Ethyl-(4-nitro-3-methylphenyl)-ether, $C_6H_3(CH_3)(NO_2)OC_2H_5$; mw 181.18, N7.73%; prisms (pet eth), mp 55°. Prepd from ethyl iodide in eth and the silver salt of 6-nitro m-cresol

Refs: 1)Beil **6**, 385 2)W.Staedel & A.Kolb, Ann **259**, 224 (1890)

Ethyl-(6-nitro-3-methylphenyl)-ether, $C_6H_3(CH_3)(NO_2)OC_2H_5$; mw 181.18, N7.73%; ndls (alc), mp 54°. Prepd by adding ethyl m-tolyl ether in HAc to sp gr 1.505 HNO_3 in the cold

Refs: 1)Beil **6**, 386 2)W.Staedel, Ann **217**, 161 (1883)

Ethyl-(4,6-dinitro-3-methyl phenyl)-ether, $C_6H_2(CH_3)(NO_2)_2OC_2H_5$; mw 226.18, N 12.39%; wh ndls (dil alc), mp 97°, sol in eth, chlf & benz. Prepd by reacting the 4-nitro-3-methyl-1-ethoxy-benzene with fuming nitric acid
Refs: 1) Beil **6**, 387, (194) 2) W. Staedel & A. Kolb, Ann **259**, 219 (1890)

Ethyl-(2,4,6-trinitro-3-methylphenyl)-ether, $C_6H(CH_3)(NO_2)_3OC_2H_5$; mw 271.18, N 15.49%; wh plates (alc), mp 75°, sol in alc, eth, benz, chlf & acet. Prepd by reacting ethyl iodide in alc with the silver salt of 2,4,6-Trinitro-m-cresol
Refs: 1) Beil **6**, 388 & (195) 2) E. Noelting & V. Salis, Ber **15**, 1864 (1881) 3) M. Giua, Gazz **49**, II, 158-66 (1919) & CA **14**, 1532 (1929) (Prepn)

5-Ethyl-2-methyl Pyridine, 2-Methyl-5-ethyl-Pyridine (MEP) or Aldehydine (2,5), $(CH_3)C_5H_3N(C_2H_5)$; mw 121.18; liquid, sp gr 0.921 at 20/20°, fr p -70.3°, bp 178.3°, n_D 1.4970 at 20°, fl p (Cleveland open cup) 165°F; nearly insol in w; sl sol in aq alc, sol in alc, eth & H_2SO_4. Can be prepd by treating paraldehyde with ammonia under high pressure and in presence of Amm acetate as a catalyst. Used as intermediate for germicides and textile finishes; as a corrosion inhibitor for chlorinated solvents (Ref 3)
Note: It has been reported that when a mixture of EtMe-pyridine with 70% nitric acid (total vol 6.2 ml) was heated in a 1-liter autoclave a sudden rise of pressure, followed by a violent explosion took place. It is advised that reaction vessels be only 1/5th full and provided with adequate cooling and fast venting
Refs: 1) Beil, **20**, 248, (86) & [162] 2) G. Krämer, Ber **3**, 262 (1870) 3) CondChemDict (1961), 739-R & 740-L (2-Methyl-5-ethyl-pyridine) (1971), p 758-R (5-Ethyl-2-methyl Pyridine) 4) Sax (1968), p 574-L

7-Ethyl-2-methyl-4-undecanol, 1-Tetradecanol or Myristyl Alcohol, $C_4H_9.CH(C_2H_5).C_2H_4.CH(OH).CH_2.CH(CH_3)$; mw 214.40, liq or wh solid, sp gr 0.8355 at 20/20°, fr p 38°, bp 264°, fl p 285°C; insol in w. Can be prepd by slow addition of a mixture of 4-methyl-2-pentanol, Zn dust and zinc oxide to sodium -2-ethyl-1-hexanolate at 175°. Used as an intermediate for synthetic lubricants, defoamers and surfactants (Ref 3)
Refs: 1) Beil, not found 2) C.A. Carter, USP 2457866 (1949) & CA **43**, 3438 (1949) 3) CondChemDict (1961), 474-R; (1971), p 372-L (7-Ethyl-2-methyl-4-undecanol), p 600-L (Myristyl Alcohol) & p 855-R (Tetradecanol) 4) Sax, 8th edit (1968), p 1143-L (Tetradecanol)

Ethylnitramine. See Nitraminoethane or N-Nitroethylamine in Vol 1 of Encycl, p A199-R, under AMINOETHANE AND DERIVATIVES

1-(N-Ethyl)nitramino-2-ethanol Nitrate or EtNENA. See under Ethylaminoethanol in this Vol

Ethyl Nitrate. See Ethane Nitrate in this Vol

Ethyl Nitrate, Azide. See Ethane Azidonitrate or Ethylazido-Nitrate in this Vol

Ethyl Nitrite. See Ethane Nitrite in this Vol

Ethyl Nitro-Nitroso-Dinitro-etc Compounds are described under Ethane in this Vol

2-Ethyl-2-nitro-1,3-propanediol Dinitrate. See 1.1-Di(methylol)-1-nitropropane Dinitrate in Vol **5**, of Encycl, p D1359-L

Ethylnitrolic Acid, $CH_3C(:NOH).NO_2$; mw 104.07, N 26.91%, OB to CO_2 -46.2%; crysts, mp 88° (decompd), sol in w, alc or eth. Can be prepd by reacting HNO_3 with the sodium salt of nitroethane
Refs: 1) Beil **2**, 189, (86) & [185] 2) V. Meyer, Ann **175**, 98 (1875)

Ethylnitrosolic Acid, $CH_3.C(:NOH).NO$; mw 88.07, N 31.79%. Prepd by reducing ethyl nitrolic acid with NaHg. Known only as its salts, as it can not be isolated from

soln. The **K salt** is prepd from acetoxyamidoxime and methanolic KOH, deep blue flakes, expl 207°; the **Ag salt** is prepd from $AgNO_3$ and the NH_4 salt soln of the acid, brn cryst, mp 120° dec, expl when heated on platinum

Refs: 1)Beil **2**, 189 2)H.Wieland, Ann **353**, 90-3(1907) & CA **1**, 2239 (1907) 3) J.Armand, CR **258** (1),207-10 (1964) & CA **60**, 10498 (1964)

Ethylolamine, Dinitro. See under Aminoethanol in Vol **1**, p A201-R

Ethyl Oleate, $C_{17}H_{33}.COOC_2H_5$; mw 310.50, lt yel oily liq, sp gr 0.869 at 20/4°, fr p <-15° (Lange), approx -32° (Ref 3), n_D 1.45189 at 20°, fl p 347.5°F (175.3°C); insol in w; sol in alc & eth. Can be prepd from oleic acid, alc and concd H_2SO_4. Used as solvent, lubricant, plasticizer or water-resistant agent (Ref 3)

Refs: 1)Beil **2**, 467, (203) & [438] 2)A. Laurent, AnnChim(Paris) [2] **65**, 298(1837) 3) CondChemDict (1961), 475-L; 8th edit (1971), 372-R 4) Sax, 3rd edit (1968), p 760-L

Ethyl Oxalate or Diethyl Oxalate, $(COOC_2H_5)_2$; mw 146.14, col, unstable aromatic liquid; combustible but not flammable; sp gr 1.079 at 20/4° (Lange), 1.09 at 20/20° (Ref 3), fr p -40.6°, bp 185.4-186°, fl p 168°F; v sl sol in w with gradual decompn; miscible with alc, eth, eth acetate and other common org solvents. Can be prepd by standard esterification procedure using ethanol & oxalic acid. The final purification, however calls for specific technique and equipment (Ref 3). Used as solvent for cellulose esters & ethers and for synthetic resins; also for radio tube cathode fixing lacquers, pharmaceuticals, etc

Refs: 1)Beil **2**, 535, (232) & [504] 2)J.Liebig, Ann **65**, 350 (1848) 3)CondChemDict (1961), 475-L & R; 8th edit (1971), 373-L 4)Sax, 3rd edit (1968), 760-L

Ethyl Oxamate or Oxamaethane, $H_2N.CO.COOC_2H_5$; mw 117.10, N11.96%; rhom plates (hot alc), mp 114-115°; sol in w & eth; v sl sol in alc. Can be prepd from diethyloxalate and ammonia gas

Refs: 1)Beil **2**, 544,(236) & [509] 2) J. Dumas & P. Boullay, AnnChim(Paris) [2] **37**, 38(1828)

Ethyloxamide and Derivatives

Ethyloxamide, $C_2H_5.NH.CO.CO.NH_2$, mw 116.12, N24.13%; ndls (w), mp 202-3° (subl below mp), sol in eth, hot w, & hot alc. Prepd from ethylamine and $C_2H_5O_2CCONH_2$ (See below) (Compare with Diethyloxamide in Vol **5** of Encycl, p D1246-L)

Refs: 1)Beil **4**, 112 & (352) 2)O.Wallach, Ann **184**, 65 (1877)

Dinitroethyloxamide, $C_2H_5.N(NO_2).CO.CO.NH(NO_2)$; mw 206.12, N27.18%, OB to CO_2 -38.8%, crysts. Was prepd in Germany (Ref 2) by nitration of ethyl oxamide with concd nitric acid

It is a powerful expl, insensitive to mechanical action; was proposed in Germany for filling shells and as an ingredient of smokeless propellants and commercial expls

Refs: 1)Beil, not found 2)Ph.Naoûm, Ger P499403 (1928) 3)CA, not found

Ethyl Oxide. Same as Ether (qv)

Ethyl Palmitate, $C_{15}H_{31}COO.C_2H_5$; mw 284.47; col ndls, sp gr 0.858 at 25/4°, mp 24-25°, bp 191° at 10 mm, n_D 1.4278 at 50°; insol in w, sol in alc & eth. Can be prepd by heating palmitic acid with ethanol in presence of HCl (Refs 1&2)

It was proposed by the duPont Co(Ref 3) to prepare flashless smokeless proplnts from NC & DNT, in which is incorporated ethyl palmitate or other esters of organic acids insol in water but forming a gelatinous mass with NC & DNT at about 80°

Refs: 1)Beil **2**, 372,(166) & [336] 2) Holzmann,ArchderPharm **236**, 440(1898) 3)DuPont&Co, Ger P567878 (1930) & CA **27**, 2814 (1933)

Ethylpentane and Derivatives

3-Ethylpentane or Triethylmethane,
$H_3C.CH_2.CH(C_2H_5).CH_2.CH_3$; mw 100.21; col liq, sp gr 0.69181 at 20°, fr p -118.6°; bp 93.5°, n_D 1.3934 at 20°; insol in w; sol in alc. Can be prepd from ethyl orthoformate, ethyl zinc, and sodium. Used in organic synthesis
Refs: 1)Beil 1, 157, (58) & [119], 2)A.Ladenburg, Ber 5, 752 (1872) 3)Cond-ChemDict (1961), 475-R; 8th edit (1971), 373-L

3-Nitro-3-ethylpentane,
$H_3C.CH_2C(NO_2)(C_2H_5).CH_2.CH_3$; mw 145.20, N9.65%; liq, sp gr at 0/0° 0.9549, bp 185-90°. Prepd from trichloronitromethane and diethyl zinc
Refs: 1)Beil 1, 157 2)J.Bewad, JPRCh [2] **48**, 377 (1893)

2,2-Dinitro-3-Ethylpentane,
$H_3C.C(NO_2)_2.CH(C_2H_5).CH_2.CH_3$
mw 190.20, N14.73%, bm yel oil, bp 211-19°. Prepd by oxidation with CrO_3/HAc of asymmetrical diethyl(propylpseudonitrol)
Refs: 1)Beil 1, 157 2)G.Born, Ber **29**, 100 (1896)

2,2,3-Trinitro-3-Ethylpentane,
$H_3C.C(NO_2)_2.C(NO_2)(C_2H_5).CH_2.CH_3$
mw 235.20, N17.87%, crysts, mp 85.5-87.5°. Prepd from the parent and ethyl nitrite in HCl to give 2-nitroso-3-chloro-3-ethylpentane, which was treated with concd HNO_3. The compd is an expl comparable in sensitivity with TNT but only 82% as powerful by Ballistic Mortor (Ref 2,3)
Refs: 1)Beil, not found 2)Bureau of Mines, High Explosives Research Division Technical Report No **25**, (1942) 3)A.H.Blatt, "Compilation of Data on Organic Explosives," OSRD Rept **2014** (1944)

Ethylpentanoate and Derivatives

Ethylpentanoate,
$H_3C.CH_2.OOC.CH_2.CH_2.CH_2.CH_3$; mw 130.19, liq, sp gr 0.8765 at 20°, bp 145.5°, n_D 1.4044 at 15°. Prepd by reducing ethyl levulinate with zinc amalgam in alcoholic HCl
Refs: 1)Beil **2**, 301,(130) & [266] 2)W. Steinkopf & A. Wolfram, Ann **430**, 142 (1923)

Ethyl-2-azidopentanoate,
$H_3C.CH_2.OOC.CH(N_3).CH_2.CH_2.CH_3$; mw 171.20, N24.55%, liq, bp 65-70°(2.8 mm Hg). Prepd by reacting NaN_3 with ethyl-2-bromopentanoate in aq alc
Refs: 1)Beil, not found 2)R.M.Moriarty & M. Rayman, Tetrahedron **21**(10), 2877-91 (1965) & CA **64**, 3312(1966)

Ethyl-5-azidopentanoate,
$H_3C.CH_2.OOC.CH_2.CH_2.CH_2.CH_2N_3$; mw 171.20, N24.55%; liq, bp 105-7° (13mm Hg). Prepd by reacting NaN_3 with ethyl-5-bromopentanoate
Refs: Beil, not found 2)D.H.R.Barton & L.R.Morgan Jr, JChemSoc **1962**, 622-31 & CA **57**, 910 (1962)

Ethyl-2-nitropentanoate
$H_3C.CH_2.OOC.CH(NO_2).CH_2.CH_2.CH_3$; mw 175.19, N8.00%; liq, sp gr 1.0713 at 20°, bp 51°(0.1 mm Hg) 90°(8mm Hg), n_D 1.4270 at 20°, sol in alc, eth, benz. Prepd from the **2-nitroso** compd by oxidizing with H_2O_2 in H_2SO_4
Refs: 1)Beil **2**, (132) 2)J.Schmidt & H. Dieterle, Ann **377**, 50 (1910) 3)W.Biernacki & T.Urbański, BullAcadPolonSci, Ser SciChim **13**(5), 349-54 (1965) & CA **63**, 16209(1965) (Prepn and infrared spectrum)

Ethyl-3-nitropentanoate,
$H_3C.CH_2.OOC.CH_2.CH(NO_2).CH_2.CH_3$; mw175.19, N8.00%; liq, bp 105-10°(15mm, contains 12% 3-bromo compd). Prepd from the reaction of $NaNO_2$ with the 3-bromo compd in the presence of phloroglucinol in dimethylformamide at 60°
Refs: 1)Beil, not found 2)W.Biernacki & T. Urbański, BullAcadPolonSci, SerSciChim **13**(5),.349-54(1965) & CA **63**, 16209 (1965) (Also infrared spectrum)

Ethyl-4-nitropentanoate,
$H_3C.CH_2.OOC.CH_2CH_2.CH(NO_2).CH_3$; mw 175.19, N8.00%; liq, sp gr 1.0955 at 20/4°, bp 68-71°(1 mm Hg, Ref 4), 94-5° (1-2 mm, Ref 2,3), n_D1.4315 at 20°. Prepd by reacting nitroethane with ethyl acrylate in aq ammonia at -50°
Refs: 1)Beil; not found 2)S. Wakamatsu & K.Shimo, JOC **27**, 1609-11 (1962) & CA **57**, 2050(1962) 3)ibid, Japan P3419 (1964) & CA **61**, 2978 (1964) 4)W.Biernacki & T.Urbański, BullAcadPolonSci, SerSciChim **13** (5), 349-54 (1965) & CA **63**, 16209 (1965) (Infrared spectrum)

Ethyl 5-nitropentanoate,
$H_3C.CH_2.OOC.CH_2.CH_2.CH_2.CH_2NO_2$; mw 175.19, N8.00%; liq, sp gr 1.075 at 25°, bp 250-5°(dec) 80°(0.8 mm Hg), n_D1.4344 at 25°. Prepd by heating $NaNO_2$ in w at 85° with $HO_2CCH(Br)(CH_2)_3CO_2C_2H_5$
Refs: 1)Beil, not found 2)W.Treibs & H.Reinheckel, Ber **87**, 341-5 (1954) & CA **49**, 4526 (1955)

Ethyl 4,4-dinitropentanoate
$H_3C.CH_2.OOC.CH_2.CH_2.C(NO_2)_2.CH_3$; mw 220.18, N12.72%. Presumably prepd by the addition of 1,1-dinitroethane to ethyl acrylate. The only refs to this compd in the open literature are a method of analysis for an expl containing it (Ref 2), and its use in an extrudable expl (Ref 3)
Refs: 1)Beil, not found 2)W.Selig, USAtom Energy Comm **UCRL-7873**, 10-19(1964) & CA **64**, 9497 (1966) 3)M.Finger, et al, USP 3480490 (1969) & CA **72**, 33910 (1970)

2,2,2-Trinitroethyl-4,4-dinitropentanoate (TNEDNP),
$(O_2N)_3C.CH_2.OOC.CH_2.CH_2.C(NO_2)_2.CH_3$; mw 355.18, N19.72%, OB to CO_2-29.3%, cryst, sp gr 1.60, mp 92.5-93°, ignition temp 297°. Was prepd at NAVORD lab by heating the acid chloride of ethylpentanoate with Trinitroethanol at 70° in presence of anhydrous $AlCl_3$. The acid chloride was prepd by the addn of 1,1-Dinitroethane to methylacrylate, followed by hydrolysis with aq HCl. Its impact sensitivity is comparable to Comp A and vacuum stability at 100°-1.1 ml gas evolved per 1g in 48 hours
Refs: 1)Beil, not found 2)W.F.Sager & D.V.Sickman, NAVORD Rept **483** (1952) 3)D.Jensen, NAVORD Rept **2498**, White Oak, Md (1952) 4)M.H.Gold & K.Klager, Tetrahedron **19**, Suppl 1,77-84 (1963) & CA **59**, 12627 (1963) (Prepn) 5)J.M.Rosen et al, Microscope **19** (2), 151-6 (1963) & CA **75**, 41551 (1971) (Cryst props)

Ethyl Perchlorate, $C_2H_5.ClO_4$; mw 128.52; oily liq, bp 89-97°(when heated covered with water). Can be prepd by distilling Ba perchlorate with Ba ethylsulfonate (Refs 1 & 2)
When dry, it is an extremely violent and sensitive expl, exploding even on pouring from one container into another (Refs 1 & 2). Its power by Trauzl Test is 110%PA (Ref 4)
Refs: 1)Beil **1**, 324, [325] & {1314} **2)** H.E.Roscoe, Ann **124**, 124(1862) 3)J.Meyer & W.Spormann, ZAnorgAllgamChem **228**, 341-51 (1936) & CA **30**, 8057 (1936) 4) Blatt, OSRD **2014**, (1944)

Ethyl Peroxide. See Diethyl Peroxide in Vol5 of Encycl p D1246-R

Ethyl Peroxide of Berthelot. See Ethyl Ozonide or Tetraethyl Ozonide

Ethyl Peroxyacetate, $CH_3CO\text{-}OO\text{-}C_2H_5$, mw 104.11, oil. Believed to have been prepd from acetic anhydride and a dilute soln of ethylhydroperoxide (Ref 2) but not further examined
Refs: 1)Beil, not found 2)A.Baeyer & V.Villiger, Ber **34**, 746 (1901) 3)Tobolsky & Mesrobian (1954), 168

ETHYLPHENOLS AND DERIVATIVES

Ethylphenols, $C_2H_5.C_6H_4OH$; mw 122.16. Three isomers are known:
o-Ethylphenol or Phlorol, col liq, sp gr 1.018 at 25/25°, fr p -45°, bp 202° at 756 mm; v sl sol in w; miscible with alc ether. Prepn is in Ref 1
m-Ethylphenol, col liq, sp gr 1.001 at 25/25°, fr p -4°, bp 214° at 752 mm; v sl sol in w; miscible with alc & eth. Prepn is in Ref 2
p-Ethylphenol, col ndls, mp 46-47°, bp 219°, v sl sol in w; v sol in alc or eth. Prepn is in Ref 3

Refs: 1)Beil **4**, 470, (234) & [442] 2)Beil **4**, 471 & [473] 3)Beil **4**, 472,(234) & [443]

Ethyl mono-nitrophenols. Beil (Ref 1) lists only one compd of undedetermined structure, yel oil, bp 212-15°, with a **Ba salt** which expl on heating. Prepd by nitration of ethylbenzene followed by treatment with nitrous acid
Refs: 1)Beil **6**, 475 2)W.Suida & S.Plohn, Monatsh **1**, 181 (1880)
Other sources list, however:

o-Ethyl 4-nitrophenol, $C_2H_5C_6H_3OH(NO_2)$; mw 167.16, ndl, mp 79-80°, sol usual organic solv. Prepd by reacting 2-pentanone with sodium nitromalonaldehyde in alcoholic base
Refs: 1)Beil, not found 2)E.C.S.Jones & J. Kenner, JChemSoc **1931**, 1850-1 & CA **25**, 5409 (1931)

o-Ethyl 5-nitrophenol, $C_2H_5C_6H_3OH(NO_2)$, mw 167.16, solid, mp 76-7° (pet eth). Prepd by treating o-ethyl 5-nitroaniline with nitrous acid at 0-5°
Refs: 1)Beil, not found 2)Y-S.Kao & P-C. Pan, Hua HsüehHsüehPao **24**, 240-53 (1958) & CA **53**, 12228 (1959)

p-Ethyl 3-nitrophenol, $C_2H_5C_6H_3OH(NO_2)$, mw 167.16, liq bp 123-5° (0.1 mm Hg), n_D 1.5705 at 25°, Prepd by treating with nitrous acid at 0° p-ethyl 3-nitroaniline
Refs: 1)Beil, not found 2)F.Wessely, et al, Monatsh **83**, 24-30 (1952) & CA **46**, 11151 (1952) 3)C. Hansch, et al, JOC **21**, 265-70 (1956) & CA **50**, 16742 (1956) (Prepn and props)

Ethyl dinitrophenols. Beil (Ref 1) lists only one compd of undetermined structure, with an expl **Pb salt**. Prepd by nitrating o-ethylphenol with cold fuming HNO_3
Refs: 1)Beil **6**, 471 2)W.Suida & S.Plohn, Monatsh **1**, 181 (1880)
Other sources list, however:

o-Ethyl 4,6-dinitrophenol, $C_2H_5C_6H_2OH(NO_2)_2$, mw 212.16, N13.20%, cryst, mp 37° bp 120° (0.01 mm Hg). Prepd by sulfonating o-ethylphenol followed by nitration starting at 0°, ending with heating. Ref (4) describes an undetermined ethyl dinitrophenol derived from ethylbenzene which has the proper mp for this isomer, indicated to decompose at 270° and having a **Na salt** exploding at 240° and a **Pb salt** exploding at 210°
Refs: 1)Beil, not found 2)J.D.Pollard, USP 2048168 (1936) & CA **30**, 6009 (1936) 3)G.G.S.Dutton, et al, CanJChem **31**, 837-41 (1953) & CA **48**, 11382 (1954) (Ref for the bp) 4)C-C.Shen, et al, Yao Hsüeh Hsüeh Pao **6**, 207-9 (1958) & CA **53**, 9126 (1959)

m-Ethyl 4,6-dinitrophenol, $C_2H_5C_6H_2OH(NO_2)_2$, mw 212.16, N13.20%, solid, mp 85°, Prepd by the mixed acid nitration of m-ethylphenol starting at 0° and ending at 100°
Refs: 1)Beil, not found 2)A.B.Sen & R.C.Sharma, JIndChemSoc **34**, 877-81(1957) & CA **52**, 13734 (1958)

p-Ethyl 2,6-dinitrophenol, $C_2H_5C_6H_2OH(NO_2)_2$, mw 212.16, N13.20%, liq, bp 130° (0.01 mm Hg). Prepd by nitration in HAc of p-ethylphenol
Refs: 1) Beil, not found 2) G.G.S. Dutton et al, CanJChem **31**, 837-41 (1953) & CA **48**, 11382 (1954)

m-Ethyl 2,4,6-trinitrophenol or 3-Ethyl Picric Acid, $C_2H_5C_6HOH(NO_2)_3$, mw 257.16, N16.34%, cryst, mp 88°. Prepd by addition of HNO_3 to 3-ethyl phenol dissolved in H_2SO_4, at 10-20°
Refs: 1)Beil, not found 2)C.E.Moore & R.Peck, JOC **20**, 673 (1955) & CA **50**, 2290 (1956)

Ethylphenylacetic Acid and Derivatives, or **Ethylbenzylate and Derivatives**
Ethylphenylacetic Acid, $C_6H_5.CH_2.COOC_2H_5$, mw 164.20, col liq, sp gr 1.033 at 20/4°, bp 227°, n_D 1.499 at 18.5°, insol in w; miscible with alc or eth. Can be prepd by simple esterification with HCl
Refs: 1)Beil **9**, 434,(173) & [297] 2) B. Radziszewski, Ber **2**, 208 (1869)

Ethylphenyl Azidoacetic Acid or *Ethylphenyl Triazoacetate,* $C_6H_5CH(N_3)COOC_2H_5$, mw 205.21, liq, sp gr 1.434 at 20/20°, bp 141° (12 mm Hg), Prepd by esterification of the azido acid with alcoholic sulfuric acid. De-

composes vigorously with concd sulfuric acid
Refs: 1)Beil **9**,(186) 2)A.Darapsky, JPrCh [2] **99**, 227 (1919)

2-Nitro Ethylphenylacetic Acid,
$O_2NC_6H_4.CH_2COOC_2H_5$, mw 209.20, ndl (alc) mp 69°, sol in usual org solv. Prepd by simple esterification with alcoholic HCl

Refs: 1)Beil **9**, 455 & (182) 2)A.Reissert & J.Scherk, Ber **31**, 395 (1898)

3-Nitro Ethylphenlacetic Acid,
$O_2NC_6H_4CH_2COOC_2H_5$, mw 209.20, oil, bp 89°(12 mm Hg). Prepd from the acid chloride and alc. Ref (2) indicates the compd is unstable
Refs: 1)Beil **9**[312] 2) J.B. Muenzen, et al, JBiolChem **68**, 504 (1926)

4-Nitro Ethylphenylacetic Acid,
$O_2NC_6H_4CH_2COOC_2H_5$, mw 209.20, pl (pet eth), mp 65.5-66°, bp 196-7°(20 mm Hg), sol alc, eth. Prepd by simple esterification with alcoholic HCl
Refs: 1)Beil **9**, 456,(183) & [312] 2)T.Maxwell, Ber **12**, 1767 (1879)

Ethylphenyl nitroacetate,
$C_6H_5CH(NO_2)COOC_2H_5$ or $C_6H_5C(:NO_2H)COOC_2H_5$, mw 209.20, oil, dec on distillation. Prepd as the **Na salt** by treating ethyl phenyl acetate with ethyl nitrate in eth in the presence of Na. A **Li salt** crystallizes from alc as ndls with one molecule of alc of crystallization; the alc evaporates at 150° and the remainder melts at 200°
Refs: 1)Beil **9**, 457 & [313] 2) W. Wislicenus & A.Endres, Ber **35**, 1755(1902)

2,4-Dinitro Ethylphenylacetate,
$(O_2N)_2C_6H_3CH_2COOC_2H_5$, mw 254.20, ndl (w), mp 35°, sol in the usual org solv. Prepd by esterification of the acid
Refs: 1)Beil **9**, 459 2)S.Gabriel & R.Meyer, Ber **14**, 824 (1881)

Ethylphenyl Acetylene or 1-Phenyl-butyne-1,
$C_2H_5.C{:}C.C_6H_5$; mw 130.19, liquid, sp gr 0.923 at 21°, bp 201-203°, insol in w, sol in alc or eth. Can be prepd from sodium phenylacetylide and ethyl iodide at 120-140°

Refs: 1)Beil **5**, 517 & [413] 2)T.M.Morgan, JChemSoc **29**, 162 (1876)

Ethylphenyl Acrylate, See Ethylcinnamate

ETHYLPHENYLAMINES AND DERIVATIVES

Ethylphenylamines or Aminoethylbenzenes, also known as *o-m-and p-Ethylanilines,* $C_2H_5.C_6H_4.NH_2$. (Not to be confused with N-Ethylanilines), mw 121.18. All three isomers are described in the literature
Refs: 1)Beil **12**, 1089, (468) & [584],(ortho) 2)Beil **12**, 1090 & (468) (meta) 3)Beil **12**, 1090 (469) & [584] (para)

2-Ethyl-4-Nitrophenylamine or 2-Ethyl-4-Nitroaniline, $C_2H_5C_6H_3(NO_2)NH_2$; mw 166.18, N16.86%; lfts or crysts, mp 87°. Prepd by nitrating with HNO_3/HAc at 50° 2-ethylacetanilide, followed by heating in acid to remove the CH_3CO-group
Refs: 1)Beil, not found 2)C.Hansch, JOC **20**, 1026-9 (1955) & CA **50**, 6368 (1956)

2-Ethyl-5-nitrophenylamine or 2-Ethyl-5-Nitroaniline, $C_2H_5C_6H_3(NO_2)NH_2$; mw 166.18, N16.86%; ndls, mp 62°. Prepd by reducing 2,4-dinitroethylbenzene with ammonium sulfide in aq alc, but not isolated (Ref 2) until later (Ref 3)
Refs: 1)Beil **12**, [584] 2)E.L.Cline & E E. Reid,JACS **49**, 3152-4 (1927) 3)C.Hansch, JOC **20**, 1026-9 (1955) & CA **50**, 6368 (1956)

2-Ethyl-6-nitrophenylamine or 2-Ethyl-6-Nitroaniline, $C_2H_5C_6H_3(NO_2)NH_2$; mw 166.18, N16.86%; crysts, mp 32°, bp 151-3° (6 mm Hg), Prepd by nitrating 2-ethylacetanilide in HAc/Ac_2O, followed by removal of the CH_3CO-with acid

Refs: 1)Beil, not found 2)L.J.Sargent, JOC **19**, 599-607 (1954) & CA **49**, 5478 (1955)

2-Ethyl-2-nitrophenylamine or 3-Ethyl-2-Nitroaniline, $C_2H_5C_6H_3(NO_2)NH_2$; mw 166.18, N16.86%; orn-red crysts, mp 32-3°. Prepd by reducing 2,3-dinitroethylbenzene with stannous chloride in ethanolic HCl
Refs: 1)Beil, not found 2)H.Kondo & S. Uyeo, Ber **70B**, 1087-93 (1937) & CA **31**, 4981 (1937)

4-Ethyl-2-nitrophenylamine or 4-Ethyl-2-Nitroaniline, $C_2H_5C_6H_3(NO_2)NH_2$; mw 166.18, N16.86%; orn prisms, mp 47-8.5°, bp 126-30°(0.4 mm Hg), subl 100°(0.3 mm Hg). Prepd by treating 3-nitro-4-bromoethylbenzene with ethanolic NH_3 at 150°
Refs: 1)Beil, not found 2)P.Karrer et al, HelvChimActa **17**, 516-23(1934) & CA **29**, 1824 (1935) 3)J.P.Lambooy & E.E.Haley, JACS **74**, 1087-9 (1952) & CA **47**, 12278 (1953)

4-Ethyl-3-nitrophenylamine, or *4-Ethyl-3-Nitroaniline,* $C_2H_5C_6H_3(NO_2)NH_2$; mw 166.18, crysts, mp 44-5°. Prepd by reducing 2,4-dinitroethylbenzene with ammonium sulfide
Refs: 1)Beil, not found 2)C.Hansch, et al, JOC **21**, 265-70 (1956) & CA **50**, 16742 (1956)

2-Ethyl-4,6-dinitrophenylamine or 2-Ethyl-4,6-dinitroaniline, $C_2H_5C_6H_2(NO_2)_2NH_2$; mw 211.18, N19.90%; crysts, mp 136°. Prepd by sulfonating 2-ethylphenyl, nitrating in the 4,6 positions, conversion to the anisole with dimethylsulfate in aniline, followed by reaction with ammonia in methanol
Refs: 1)Beil, not found 2)P.J.C.Fierens, et al, BullSocChimBelges **64**, 658-66 (1955) & CA **50**, 11260 (1956)

4-Ethyl-2,3-dinitrophenylamine or 4-Ethyl-2,3-dinitroaniline, $C_2H_5C_6H_2(NO_2)_2NH_2$; mw 211.18, N19.90%; yel ndls (dil alc), mp 121.6°. Prepd by heating the corresponding acetanilide in sulfuric acid
Refs: 1)Beil **12**, [585] 2)O.L.Brady et al, JCS **1928**, 981 & CA **22**, 2353 (1928)

4-Ethyl-2,5-dinitrophenylamine or 4-Ethyl-2,5-dinitroaniline, $C_2H_5C_6H_2(NO_2)_2NH_2$; mw 211.18, N19.90%; red prisms (alc), mp 125°. Prepd as a by product from prepn of the 2,3 compound. 4-Ethylacetanilide is mono-nitrated in the 3-position then further nitrated; the 2,3-and 2,5-(3,5-) products may be isolated and hydrolyzed to the anilines
Refs: 1)Beil **12**, [585] 2)O.L.Brady et al, JCS **1928**, 981 & CA **22**, 2353 (1928)

4-Ethyl-2,6-dinitrophenylamine or 4-Ethyl-2,6-dinitroaniline, $C_2H_5C_6H_2(NO_2)_2NH_2$; mw 211.18, N19.90%; orn-yel prisms (alc), mp 134-5°, sol in chlf, benz, alc & eth. Prepd by heating the corresponding acetanilide in HCl
Refs: 1)Beil **12**, 1092 & [585] 2)H. Paucksch, Ber **17**, 769(1884)

4-Ethyl-3,5-dinitrophenylamine or 4-Ethyl-3,5-dinitroaniline, $C_2H_5C_6H_2(NO_2)_2NH_2$; mw 211.18, N19.90%; gold ndls (alc), mp 175°, (Ref 3), yel prisms (ligroin), mp 110°(Ref 2). Prepd by the ammonium sulfide reduction of 2,4,6-trinitroethylbenzene (Refs 2 and 3; Ref 3 was aware of the mp difference)
Refs: 1)Beil **12**, 1092 & [585] 2)G.Schultz & A.Sander, Ber **42**, 2634 (1909) 3)O.L. Brady, et al, JCS **1928**, 980 & CA **22**, 2353 (1928)

N-Ethyl-N-Nitroso-2,4,6-Trinitroaniline $C_6H_2(NO_2)_3N(NO)C_2H_5$; mw 285.17, N 24.56% OB to CO_2 -70.2%; yel crysts, mp 73°. Prepd by treating N-ethyl-2,4,6-trinitroaniline with soldium nitrite in sulfuric acid. This compd is expl

Note: This compd was described in this Vol, p E207-L, under the name **N-Ethyl-2,4,6-trinitro-N-nitrosoaniline,** giving as a Ref: T.J. Nolon et al, BritP 217614(1922) & CA **19**, 300(1925)

Refs: 1)Beil, not found 2)C.A.Parker, JCS **1946**, 772-5 & CA **41**, 429 (1947)

N-Ethyl-N,2,4,6-Tetranitroaniline or **Ethyl Tetryl.** See this Vol, p E207-L. The following information was not included there:
1) Desseigne prepd by reacting ethylamine with dinitrochlorobenzene, followed by mixed acid nitration at 40-50°
2) Ethyl Tetryl forms a eutectic, mp 75.5° with Tetryl (60–70% ET / 30–40% T)
3) It is stable to Abel Test at 80°
4) Impact Sensitivity – 48% detonations with 2kg wt falling 2.5 meters

5) Initiation Sensitivity – 0.40g MF detonate 30mm diam cartridge contg 50g sample at sp gr 1.65

N-Ethyl-N,2,3,4,6-Pentanitroaniline. See this Vol, p E207-R. It is an expl compd

N-(2-Nitratoethyl)-N,2,4,6-Tetranitroaniline or Pentryl. See Vol 1, p A425-L

Ethylphenylcarbamate and Derivatives

Ethylphenylcarbamate or N-Phenylurethane, $C_6H_5.NH.COOC_2H_5$; mw 165.19, N8.48%; wh crysts with aromatic odor, sp gr 1.106 at 20/4°, mp 52-53°, bp 237°(sl decomp); insol in cold w; sol in alc, eth & boiling w. Can be prepd by the action of ethanol on phenyl isocyanate

Can be nitrated to produce expl compds

Refs: 1)Beil **12**,320, (218) & [184] 2) Wilm & Wischin,Ann **147**, 157 (1868) 3) CondChemDict (1961), 476-L

Ethyl-2-nitrophenylcarbamate, $O_2NC_6H_4.NH.COOC_2H_5$; mw 210.19, N13.33%; yel prisms (pet eth), yel ndls (alc), mp 58°, sol in benz & ligroin. Prepd by heating 2-nitroaniline with ethylchloroformate in chlf

Refs: 1)Beil **12**, 694 [372] 2)C.Rudolph, Ber **12**, 1295 (1879)

Ethyl-3-nitrophenylcarbamate, $O_2NC_6H_4NH.COOC_2H_5$; mw 210.19, N13.33%; yel ndls, mp 57°(65°), sol alc, eth, chlf, benz, acet, Prepd from 3-nitrobenzamide, bromine and sodium alcoholate

Refs: 1)Beil **12**, 706 2)E.Jeffreys, AmChemJ **22**, 41 (1899)

Ethyl-4-nitrophenylcarbamate, $O_2NC_6H_4.NH.COOC_2H_5$; mw 210.19;N13.33%; ndls(benz), mp 132°, sol in alc & benz. Prepd by treating 4-nitrobenzoic acid bromamide in alc with sodium alcoholate

Refs: 1)Beil **12**, 723 & [392] 2)S.E.Swartz, AmChemJ **19**, 301 (1897)

Ethyl-2,4-dinitrophenylcarbamate, $(O_2N)_2C_6H_3.NH.COOC_2H_5$; mw 255.19, N16.47%; yel-brn ndls (alc), mp 110-11°, sol in alc. Prepd by treating the parent with HNO_3 in the cold

Refs: 1)Beil **12**, 755,(363) & [410] 2) P.van Romburgh, RecTravChim **10**, 141 (1891)

Ethyl-3,5-dinitrophenylcarbamate, $(O_2N)_2C_6H_3.NH.COOC_2H_5$; mw 255.19, N16.47%; ndls, mp 83°. Prepd by reacting phosgene with 3,5-dinitroaniline to give 3,5-dinitrophenyl isocyanate, which was reacted with alc

Refs: 1)Beil, not found 2)J.J.Blanksma & G.Verberg, RecTravChim **53**, 1037-46 (1934) & CA **29**, 463 (1935)

Ethyl-N-nitro-3,5-dinitrophenylcarbamate, $(O_2N)_2C_6H_3N(NO_2).COOC_2H_5$; mw 300.19, N18.68%, OB to CO_2-74.7%, col ndls, mp 102°. Was prepd by nitrating 3,5-Dinitrophenylcarbamate with absol nitric acid at -15°, a small amt of a by-product was produced at the same time (mp 118°).

Explosive props were not examined

Refs: 1)Beil, not found 2)J.J.Blanksma & G.Verberg, Rec **53**, 1042-43 (1934) & CA **29**, 469 (1935)

Ethyl-2,4,6-trinitrophenylcarbamate or **Picrylurethane** $(O_2N)_3C_6H_2.NH.COOC_2H_5$; mw 300.19, N18.67%, OB to CO_2-74.7%; ndls (dil alc) or plates (concd alc), mp 147°. Prepd from the parent or the 2,4-dinitro derivative by warming with sp gr 1.5 HNO_3, or by treating with mixed acid, cold. This compd is expl

Refs: 1)Beil **12**, 768,(370) & [423] 2)P. van Romburgh, RecTravChim **10**, 142 (1891)

Ethyl-2,3,5-trinitrophenylcarbamate, $(O_2N)_3.C_6H_2.NH.COOC_2H_5$; mw 300.19, N18.67%, OB to CO_2-74.7%, yel lfts, mp 118° Was prepd by nitrating Ethyl-3,5-dinitrophenylcarbamate with mixed nitric-sulfuric acid at -15°, a small amt of a by-product isomer melting at 144° was obtd at the same time; the by-product was not identified. This compd is expl

Refs: 1)Beil, not found 2)J.J.Blanksma & G.Verberg, Rec **53**, 1044-45 (1934) & CA **29**, 463 (1935)

Ethylphenylcarbamate, Ethyl Ester or Ethyl-Phenylurethane. (Äthylphenylcarbamidsäure-

äthylester, in Ger), $C_6H_5.N(C_2H_5).COOC_2H_5$; mw 193.25, N 7.25%; col oil, sp gr 1.0436 at 20/4°, bp 130° at 14mm or 137.5-138° at 19mm, n_D 1.50573 at 20°; solubilities are given in Ref 3. Can be prepd from ethylaniline, ethylchloroformate and sodium bicarbonate in ice water

Tavernier (Ref 4) reported its heat of combustion as 7561 cal/g at C_v, heat of formaformation 409 cal/g at C_v and 437 at C_p. It was used by the Germans, especially in Nitroguanidine propellants

Refs: 1)Beil **12**, 422 & [237] 2)O.Schmidt, ZPhysikChem **58**, 518(1907) 3) R. Dalbert & J. Tranchant, MP **30**, 338(1948) 4) P. Tavemier, MP **38**, 306 & 328(1956) & CA **51**, 15952(1957)

Ethylphenylglycinate and Derivatives

Ethylphenylglycinate or N-Phenylglycine Ethylester, $C_6H_5NH.CH_2COO.C_2H_5$; mw 179.21, N 7.82%; lfts, mp 57–58°, bp 273° (sl decompn); v sl sol in hot w; v sol in hot alc; v sol in eth. Can be prepd by warming ethylchloroacetate with aniline

Refs: 1)Beil **12**, 470, (263) & [249] 2) P.J. Meyer, Ber **8**, 1156 (1875)

Ethyl-2-nitrophenylglycinate, $O_2N.C_6H_4.NH.CH_2.COOC_2H_5$; mw 224.21, N12.50%; yel crysts, mp 77-8°. Prepd by heating ethyl bromoacetate with 2-nitroaniline

Refs: 1)Beil, not found 2)F.E.King & J.W. Clark-Lewis JChemSoc **1951**, 3080-5 & CA **47**, 2755 (1953)

Ethyl-3-nitrophenylglycinate, $O_2NC_6H_4.NH.CH_2.COOC_2H_5$; mw 224.21, N12.50%; crysts(alc), mp 84°. Prepd by boiling 3-nitroaniline with ethylchloroacetate in a little alc

Refs: 1)Beil **12**, 709 2)E.Deutsch, JPrChem [2] **76**, 353 (1907)

Ethyl-4-nitrophenylglycinate, $O_2N.C_6H_4.NH.CH_2.COOC_2H_5$; mw 224.21, N12.50%; crysts (alc), mp 132-4°. Prepd by heating ethyl bromoacetate with 4-nitroaniline and sodium acetate

Refs: 1)Beil, not found 2)P.L.Southwick, et al, JACS **78**, 1608-11 (1956) & CA **50**, 14599 (1956)

Ethyl-2,4-dinitrophenylglycinate $(O_2N)_2C_6H_3.NH.CH_2COOC_2H_5$; mw 269-21, N15.61%; gr-yel ndls (alc), mp 144°, sol in HAc & cold acet. Prepd by heating 2,4-dinitrochlorobenzene with ethyl glycocollate hydrochloride in alkaline alc

Refs: 1)Beil **12**, (363) 2)E.Abderhalden & P.Blumberg, H-S'sZeitPhysiolChem **65**, 320 (1910) 3)S.Passeron & G.A.Brieux, BullSoc Chim Fr **1963**, 35-40 & Ca **58**, 13305 (1963)

Ethyl-2,4,6-trinitrophenylglycinate, $(O_2N)_3C_6H_2.NH.CH_2.COOC_2H_5$; mw 314.21, N17.83%. Apparently prepd by Gidaspov, (Ref 2), but no details are given in CA This compd is an expl

Refs: 1)Beil not found 2)B.V.Gidaspov, et al, ReaktSposobnostOrgSoedin **7** (4), 931-43 (1970) & CA **75**, 48096(1971)

Ethylphenylglycineazide-o-carboxylate (Phenylglycinazid-o-carbonsäure-äthylester in Ger),

C_6H_4 (ortho) $NH.CH_2.CO:N_3$ / $CO.OC_2H_5$;

mw 248.24, N22.57%; oily liquid, violently exexploding. Prepd by treating with HNO_2 the hydrazide, which came from heating hydrazine hydrate with ethyl-2-carbethoxyphenylglycinate

Refs: 1)Beil, not found 2)Th.Curtius, JPraktChem **125**, 111 & 136 (1930) & CA **24**, 3233 (1930)

Ethylphenylhydrazine and Derivatives

Two isomers are listed in Beil:

Ethylphenyldydrazine (α,α), $C_6H_5N(C_2H_5).NH_2$; mw 136.19, N20.57%; oil, sp gr 1.018 at 15°, bp 237°; soly is not given. Prepn is described in Ref 1

Ethylpenylhydrazine (α, β), $C_6H_5NH.NH.C_2H_5$, mw 136.19, N20.57%;

oil, sp gr 1.004 at 15/15°, bp 237-40° (in nitrogen), sl sol in w, v sol in alc or eth. Prepn is described in Ref2
Refs: 1)Beil **15**, 119, (27) & [50] 2) Beil **15**, 120 & (50)

1-Ethyl-1-(4-nitrophenyl)hydrazine
$O_2N.C_6H_4N(C_2H_5).NH_2$; mw 181.19, N23.19%; yel crysts (pet eth), mp 74°. Prepd by boiling with concd HCl the product from the reaction of EtI with p'-$O_2NC_6H_4NHN{:}CHC_6H_5$ (K salt)
Refs: 1)Beil, not found 2)V.R.Ciusa & G.Rastelli, Gazz **54**, 72-8 (1924) & CA **18**, 2139 (1924)

1-Ethyl-1-(2,4-dinitrophenyl) hydrazine,
$(O_2N)_2C_6H_3N(C_2H_5).NH_2$; mw 226.19, N24.77%; yel ndls (alc), mp 98-100°. Prepd by heating with concd HCl the product from the reaction of EtI with 2,4-$(O_2N)C_6H_3NH{:}N{:}C_6H_5$ (K salt). Forms a **Picrate salt**, yel ndls (50% alc), mp 90–2°
Refs: 1)Beil, not found 2)M.Ragno, Gazz **75**, 200-4(1945) & CA **41**, 4127 (1947)

Ethyl-2,4,6-trinitrophenyl-hydrazine or Ethylpicrylhydrazine (Not known if it is the 1,1-or the 1,2-isomer), $(O_2N)_3C_6H_2N_2H_2(C_2H_5)$; mw 271.19, N25.83%; yel plates (chlf), mp 200° (sl dec), sol in alc, concd HCl. Prepd from ethylhydrazine and picryl chloride in cold alc
Refs: 1)Beil **15**, 493 2)E.Fischer, Ann **199**, 299 (1879)

1-(2,2,2-Trinitroethyl)-2-(2,4-dinitrophenyl)hydrazine,
$(O_2N)_2C_6H_3.NH.NH.CH_2C(NO_2)_3$; mw 361.19 N27.15%, OB to CO_2-42.1%, crysts, mp 95-6° Prepd by heating $CH(NO_2)_3$ in chlf with the 2,4-dinitrohydrazine of formaldehyde. Nitration in Ac_2O at 10-15° gave a product, mp 133-4°, not ring nitrated, but not further examined. This compd is expl
Refs: 1)Beil, not found 2)L.S.Simonenko et al, IzvAkadNaukSSSR, SerKhim **10**, 2309-11 (1969) & CA **72**, 43044 (1970)

1-(2,2,2,-Trinitroethyl)-2-(2,4,6-trinitrophenyl) hydrazine,
$(O_2N)_3C_6H_2.NH.NH.CH_2C(NO_2)_3$; mw 406.19, N27.59%, OB to CO_2-27.6%; crysts, mp 120-1°. Prepd by heating $CH(NO_2)_3$ in chlf with the 2,4,6-trinitrophenylhydrazone of formaldehyde This compd is expl
Refs: 1)Beil, not found 2)L.S.Simonenko et al, IzvAkadNaukSSSR, Ser Khim **10**, 2309-11 (1969) & CA **72**, 43044 (1970)

Ethylphenyl Ketone or Propiophenone,
$C_2H_5.CO.C_6H_5$; mw 134.17; water-white to sl amber liq with strong odor; sp gr 1.012 at 20/20°, congealing temp 17.5-21°, n_D1.527 at 20°, fl p (TOC) 210°F; insol in w, glycol or glycerine; miscible with alc, eth, benz or toluene. Can be prepd by heating HCl with the product ($2C_9H_8$+ 3HgO+3$HgCl_2$) arising from aqueous $HgCl_2$ and α-phenylallylene
Used as starting material for prepn of ephedrine, several other pharmaceuticals and numerous organic chemicals (Ref 3)
Refs: 1)Beil **7**, 300, (159) & [231] 2) A. Körner, Ber **21**, 277 (1888) 3)CondChemDict (1961), 945-L (Propiophenone); 8th edit (1971), 733-R to 734-L (Propiophenone)

Ethylphenylureas and Derivatives
N,N'-Ethylphenylurea
$C_2H_5.NH.CO.NH.C_6H_5$; mw 164.20, N 17.06%; ndls (from aq alc), mp 99°; sol in alc Can be prepd from aniline and ethylisocyanate
Refs: 1)Beil **12**, 348, (231) & [205] 2)A. Wurtz, Répertoire de Chimie Pure **4**, 203 (1862)

N,N-Ethylphenylurea,
$C_2H_5.N(C_6H_5).CO.NH_2$; mw 164.20, N17.06%; plates (pet eth), mp 62°, v sol in w and organics. Prepd from N-ethylaniline .HCl and potassium cyanate
Refs: 1)Beil **12**, 422 & [237] 2)W.Gebhardt, Ber **17**, 2095 (1884)

N-(2-Azidoethyl)-N'-phenylurea,
$N_3CH_2CH_2NH.CO.NH.C_6H_5$; mw 205.22, N34.13%; ndls (benz), mp 99°, sol in cold alc, EtAc, chlf & hot benz. Prepd from 2-azidoethylamine and phenylisocyanate in benz
Refs: 1)Beil **12**, (231) 2)M.O.Forster & S.H.Newman, JChemSoc **99**, 1281 (1911)

N-Ethyl-N'-(2,4-dinitrophenyl)urea,
$C_2H_5NH.CO.NH.C_6H_3(NO_2)_2$; mw 254.20, N22.04%; crysts, mp 161°. Prepd by heating

N-Nitro-N′(2,4-dinitrophenyl)urea with ethylamine in alc
Refs: 1)Beil, not found 2)J.L.McVeigh & J.D.Rose, JChemSoc **1945**, 621-2 & CA **40**, 320 (1946)

N-Ethyl-N′-(3,5-dinitrophenyl)urea,
$C_2H_5NH.CO.NH.C_6H_3(NO_2)_2$; mw 254.20, N22.04%; crysts, mp 155°. Prepd by heating ethylisocyanate with 3,5-dinitroaniline at 120° in a sealed tube, or by reacting ethylamine with 3,5-dinitrophenylisocyanate
Refs: 1)Beil, not found 2)J.J.Blanksma & G.Verberg, Rec **53**, 1037-46 (1934) & CA **29**, 463 (1935)

N-Ethyl-N-nitro-N′-(2,4-dinitrophenyl)urea,
$C_2H_5N(NO_2).CO.NH.C_6H_3(NO_2)_2$; mw 299.20, N23.41%; crysts (pptd from acet with pet eth), mp 90-5° (dec), sol in acet & chlf. Prepd from the parent and 100% HNO_3 at -15°, then ambient temp
Refs: 1)Beil **12**, [411] 2)L.C.E.Kniphorst, Rec **44**, 696, 717(1925) & CA **20**, 589 (1926)

N-Ethyl-N-nitro-N′-(3,5-dinitrophenyl)urea
$C_2H_5N(NO_2).CO.NH.C_6H_3(NO_2)_2$; mw 299.20, N 23.41%; crysts, mp 98°. Prepd by treatment of N-ethyl-N′-(3,5-dinitrophenyl)urea with 100% HNO_3 at -15°
Refs: 1)Beil, not found 2)J.J.Blanksma & G.Verberg, Rec **53**, 1037-46 (1934) & CA **29**, 463 (1935)

N-(2,2,2-Trinitroethyl)-N′-phenylurea,
$(O_2N)_3CCH_2NH.CO.NH.C_6H_5$; mw 299.20, N23.41%; crysts, sp gr 1.51, mp 162°, bp ignites above 418°. Prepd by refluxing phenylurea with excess trinitroethanol in methanol for 3 hours (Ref 2). Its sensitivity to impact is about that of Comp B
Refs: 1)Beil, not found 2)D.L.Kouba & R.H.Saunders, Hercules Powder Co Progress Rept, Sept 1950, High Explosives Task A, Contract NOrd 9925 3)W.F.Sager & D.V. Sickman, NAVORD Rept **483**, (1952), p 14

N-Ethyl-N-nitro-N′-(2,4,6-trinitrophenyl)urea,
$C_2H_5N(NO_2).CO.NH.C_6H_2(NO_2)_3$; mw 344.20, N24.42%, OB to CO_2 –60.5%; crysts, mp 60-105° (dec), unstable, Prepd from the parent and mixed acid
Refs: 1)Beil **12**, [423] 2)L.C.E.Kniphorst, Rec **44**, 700 (1925) & CA **20**, 590 (1926)

N-(2,2,2-Trinitroethyl)-N′-nitro-N′-(3,4-dinitrophenyl)urea,
$(O_2N)_2C_6H_3.N(NO_2).CO.NH.CH_2.C(NO_2)_3$; mw 434.19, N25.81%; OB to CO_2 -29.5%; crysts, sp gr 1 78, mp 110°, bp ignites at above 410°. Can be prepd by nitrating N-Trinitroethyl-N′-phenylurea with mixed nitric-sulfuric acid (Ref 2). It is an expl with sensitivity to impact about that of Tetryl.
This compd may be used in pressed HE compns, but its instability on heating may prevent its use in castable mixts
Refs: 1)Beil, not found 2)D.K.Kouba & R.H.Saunders, Hercules Powder Co, Progress Rept, Sept 14,1950, High Explosives, Task A, Contract NOrd 9925 3)W.F.Sager & D.V. Sickman, NAVORD Rept **483**, (1952), 19

Ethylphenylurethane. See Ethylphenylcarbamate, Ethylester in this Vol

Ethylphosphate and Derivatives
Ethylphosphate or Triethylphosphate,
$(C_2H_5O)_3PO$; mw 182.16, col liq, sp gr 1.068 at 20/4°, fr p-56.4°, bp 215-216°, fl p 240°F (115.6°), n_D1.4055 at 20°, sol in alc, eth and in most common org solvents, sol in w- the solns are stable at ord temp, but slowly hydrolyze when hot; compatible with many gums and resins. Can be prepd by heating lead diethylphosphate
Used as high-boiling solvent; as plasticizer for resins, gums;in manuf of plasticides and as catalyst (Ref 3)
Refs: 1)Beil, **1**, 332, (167) & [331] 2) F.Vögeli, Ann **69**, 190 (1849) 3)CondChemDict (1961), 1157-L (Triethylphosphate); 8th edit (1971), 892-L

Tris-(2-nitroethyl)phosphate,
$(O_2NCH_2CH_2O)_3PO$; mw 317.15, N13.25%, OB to CO_2 -53.0%; liq, sp gr 1.752 at 30/4° bp 159-60° (3.5 mm Hg), n_D1.4471 at 25°. Prepd by reacting $POCl_3$ with 2-nitroethanol
Refs: 1)Beil, not found 2)T.Maeshima, KogyoKagakuZasshi **62**, 1853-5 (1959) & CA **57**, 16863 (1962)

Tris-(trinitroethyl)phosphate or Trinitrotriethylphosphate, $OP[OCH_2.C(NO_2)_3]_3$, mw 587.14, N21.47%; OB to CO_2+ 12.3% crysts, sp gr 1.74, mp 126°, bp-ignites at 235°; soly (?). Was prepd at Hercules Powder Co laboratory by the action of phosphorous pentachloride on Trinitroethanol(Ref 2)

It is an expl with impact sensitivity about that of Pentolite; its vacuum stability at 100° was 4.8ml gas evolved per gram in 48 hours

Refs: 1)Beil, not found 2)Hercules Powder Co,Progress Rept, Feb (1949),High Explosives, Task A, Contract NOrd 9925 3) W.F.Sager & D.V.Sickman, NAVORD Rept **483**, (1952), p 34

Ethylphosphorodimethylamidycyanadate. See GA Chemical Warfare Agent in Vol **2**, p C167-R. Originally it was German Agent of WWII, called Tabun, Trilon 83, T83 or T100, described in PATR **2510** (1958), p Ger 204-L

Ethylphthalate or Palatinol A. See o-Diethylphthalate in Vol **5** of Encycl, pD1248-L

As its nitro-compds are not listed there, we are describing them here:

3-Nitro Ethylphthalate,
$(O_2N)C_6H_3(CO_2C_2H_5)_2$; mw 267.24, N5.24%; prisms (alc), mp 45°, insol in w, sol in alc & eth. Prepd by esterification of the diacid in hot alc with HCl

Refs: 1)Beil **9**, 826, (368) & [606] 2) O. Miller, Ann **208**,243 (1881)

4-Nitro Ethylphthalate,
$(O_2N)C_6H_3(CO_2C_2H_5)_2$; mw 267.24, N5.24%; plates (alc), mp 33-4°, bp 140°(0.01 mm Hg) & 213°(17 mm Hg). Prepd from esterification of the diacid in alc with HCl

Refs: 1)Beil **9**, 831 & [607] 2)O.Miller, Ann **208**, 227, 234 (1881)

5-Nitro Ethylisophthalate,
$(O_2N)C_6H_3(CO_2C_2H_5)_2$; mw 267.24, N5.24%; ndls (alc), mp 83.5°, sl sol in w or cold alc, sol in hot alc. Prepd by refluxing the diacid in alc with HCl

Refs: 1)Beil **9**, 840 2)H.E.Storrs & R.Fittig, Ann **153**, 288 (1870)

3-Nitro Ethylterephthalate,
$(O_2N)C_6H_3(CO_2C_2H_5)_2$; mw 267.24, N5.24%; ndls (dil alc), mp 51°, sol benz,alc, eth, acet, insol w. Prepd from alc and the diacid chloride

Refs: 1)Beil **9**, [614] 2)M.A.Soderman & J.B. Johnson, JACS **47**, 1395 (1925)

4,5-Dinitro Ethylphthalate
$(O_2N)_2C_6H_2(CO_2C_2H_5)_2$; mw 312.24, N8.97%; crysts, mp 109°. Prepd from the diacid chloride by treatment with alc

Refs: 1)Beil, not found 2)H.Goldstein & J.P.Merminod, HelvChimActa **35**, 1476-80 (1952) & CA **47**, 4868 (1953)

4,6-Dinitro Ethylisophthalate,
$(O_2N)_2C_6H_2(CO_2C_2H_5)_2$; mw 312.24, N8.97%; crysts, mp 124°. Prepd by esterification of the diacid

Refs: 1)Beil, not found 2)P.Ruggli & O. Schmid, HelvChimActa **18**, 247-54 (1935) & CA**29**, 3667 (1935) 3)A.H.Blatt, JOC **25**, 2030-4 (1960) & CA **55**, 14357 (1961)

2,5-Dinitro Ethylterephthalate,
$(O_2N)_2C_6H_2(CO_2C_2H_5)_2$; mw 312.24, N8.97%; ndls (alc), mp 144°, sol in alc, eth & benz. Prepd by esterification of the diacid

Refs: 1)Beil **9**, 853 2)C.Haeussermann & E.Mertz, Ber **26**, 2984 (1893)

Bis-(Nitroethyl)-phthalate or Dinitrodiethylphthalate, $C_6H_4[COO.CH_2.CH_2(NO_2)]_2$, mw 312.24, N8.97%; liq, sp gr 1.652 at 30/3°, bp 179-82°(4 mm Hg), n_D1.5347 at 25°. Prepd by heating phthalicanhydride with 2-nitroethanol in toluene containing toluene sulfonic acid

Refs: 1)Beil, not found 2)T.Maeshima, KogyoKagakuZasshi **62**, 1853-5 (1959) & CA **57**, 16863 (1962)

Bis(Trinitroethyl)-phthalate or Hexanitrodiethyl-phthalate, $C_6H_4[COO.CH_2.C(NO_2)_3]_2$; ortho derivative; mw 492.23, N17.07%, OB to CO_2-39.0%, crysts, sp gr 1.62, mp 125°, bp-ignites at 267°. Can be prepd by heating the acid chloride of diethylphthalate with trinitroethanol (Ref 2) or from the same reactants in carbon tetrachloride at 30° in the presence of Friedel-Craft's catalysts, such as $AlCl_3$(3). It is an explosive with sensitivity to impact comparable to Tetryl

Refs: 1)Beil, not found 2)Hercules Powder Co Progr Rept April 1949, High Explosives, Task A, Contract Nord 9925 3)Marion Hill, NAVORD Rept **2245**, (NOL)(1951), Res & Devel in New Chemical Expls 4)W.F.Sager & D.V. Sickman, NAVORD Rept **483** (1952), p 16 5) G.A.Wetterholm, Ger P934694 (1955) & CA **53**, 17513 (1959) 6)M.E.Hill USP3223725 (1965) & CA **64**, 6570 (1966)

Ethylpicramide. See N-Ethyl-2,4,6-trinitroaniline, under N-Ethylaniline in this Vol

Ethyl Picrate. See 3-Ethyl-2,4,6-trinitrophenol under ETHYLPHENOLE AND DERIVATIVES in this Vol

Ethyl Picrate Nitramine. See 3-Ethyl-2,4,6-trinitrophenol Nitramine under ETHYLPHENOLE AND DERIVATIVES in this Vol

Ethylpicryl Nitramine. Same as Ethylpicrate Nitramine

Ethylpropanediol and Derivatives

2-Ethyl-1,3-propanediol or Ethyltrimethyleneglycol, $HO.CH_2.CH(C_2H_5).CH_2OH$; mw 104.15 liq, bp 124-7°(16 mm Hg). Prepd by the lithium aluminum hydride reduction of 2-ethyl diethylmalonate

Refs: 1)Beil, not found 2)S.Searles, et al, JOC **24**, 1839-44 (1959) & CA **54**, 9874 (1960)

2-Nitro-2-ethyl-1,3-propanediol (NEP), $HO.CH_2.C(NO_2)(C_2H_5)-CH_2OH$, mw 149.15, N9.39%; ndls (w), mp 57-8°, sol in w, alc & eth. Was first prepd by Pauwels (Ref 2) on condensation of formaldehyde with 1-nitropropane in presence of K carbonate. Vanderbilt & Hass (Ref 3 & 4) described methods of prepn with yields up to 96% using formaldehyde, 1-nitropropane and tertiary amines or alkali hydroxides. Médard (Ref 5) and Médard & Thomas (Ref 6) detd its heats of combustion (703.7 kcal/m,C_v) and formation. Médard also described its prepn and properties

Refs: 1)Beil **1**, 483 2)J.J.Pauwels,Rec **71**, 27 (1898) 3)B.M.Vanderbilt & H.B. Hass, USP 2139120 (1938) 4) B.M. Vanderbilt & H.B.Hass, IEC **32**, 34(1940) 5)L.Médard, MP **35**, 149-50 (1953) & CA **49**, 5843 (1955) 6)L.Médard & M.Thomas, MP **35**, 172 (1953) & CA **49**, 11284 (1955)

2-Nitro-2-ethyl-1-3-propanediol Dinitrate, $(O_2NO)CH_2.C(NO_2)(C_2H_5)CH_2(ONO_2)$, mw 239.15, N17.57%; OB to CO_2 −43.5%; pale yel liq; sp gr 1.443 at 20/20°, bp – explodes; n_D 1.4734 at 20°; sl sol in w. Was prepd by Médard (Ref 2) by nitrating technical 2-Nitro-2-ethyl-1,3-propanediol with mixed acid. For this he added in small portions, with stirring, 25g of NEP to 100g of mixed acid (HNO_3 40 & H_2SO_4 60%) precooled to 0° and allowing the temp to rise to ca 8° at the end of addition. The stirring was continued until the temp reached 15° and then the mixt was poured into 600 ml of w at ca 50°. After washing the oil, first with w and then with dil Na bicarbonate soln, it was dried in a desiccator over Ca chloride. The yield was 92% of theory

Following props are listed in Refs 2&3:

Gelatinizing Action on NC-practically none even after 8 days at 50°

Gelatinizing Action on Polyvinyl Acetate-complete

Heat of Combustion at C_v - 693.5 kcal/kg

Heat of Combustion at C_p - 691.3 kcal/kg

Heat of Formation at C_v - 82.7 kcal/kg

Heat of Formation at C_p - 88.5 kcal/kg

Impact Sensitivity - sl less sensitive than NG

Initiation Sensitivity - placed in a glass tube 17mm diam could not be initiated even by means of Briska Cup

Power by French Lead Block Test (Cup)- 125% of PA

Viscosity - 49 centipoises vs 35 cps for NG at 20°

Volatility - 10g sample left in a crystallizer at 40° for several days showed average loss of 1.08 mg per square decimeter of surface. This value is just slightly below that for NG

Uses - Several expl mixtures based on the dinitrate and AN were prepd and examined in France. Most of these mixtures were very difficult to initiate

Refs: 1)Beil, not found 2)L.Médard, MP

35, 149(1953) & CA 49, 5843(1955)
3) L. Médard & M. Thomas, MP 35, 157 & 172(1953) & CA 49, 11284(1955)

Ethylpropionate or Propionic Ester,
$C_2H_5.COOC_2H_5$; mw 102.10, col, flammable liq with odor resembling pineapple; sp gr 0.896 at 15/4°; fr p -73°, bp 99.1°, n_D 1.3844 at 20°, fl p 12°C; sl sol in w; miscible with alc & eth, Can be prepd by treating ethanol with propionic acid. Used in flavoring syrups, as solvent for cellulose ethers & esters and for various resins; also as a cutting agent for pyroxylin
Refs: 1)Beil, 2, 240 2)CondChemDict (1961), 476-L & R; 8th edit(1971), 374-L 3)Sax, 3rd edit (1968), 761-L

2-Ethyl-3-propyl-acrolein or 2-Ethylhexen-2-al, $C_3H_7.CH{:}C(C_2H_5).CHO$; mw 126.19, lt yel liq with strong unpleasant odor, sp gr 0.81518 at 20/20°, bp 175.0° at 760 mm, fl p 155°F, vapor pressure 1.0 mm at 20°, viscosity 0.113 poises at 20°. Can be prepd by shaking butyraldehyde with w and 10% NaOH, Used in org synthesis (as intermediate), insecticide and in warning agents and leak detectors
Refs: 1)Beil 1, 744 & [799] 2)G.A.Raupenstrauch, Monatsh 8, 112 (1887) 3)Cond ChemDict (1961), 476-R; 8th edit (1971), 761-R 4)Sax, 3rd edit (1968), 374-L

Ethylpropylamine and Derivatives
Ethylpropylamine,
$H_3C.CH_2.HN.CH_2.CH_2.CH_3$; mw 87.16, col liq, sp gr 0.773 at 24°, bp 79.8°(747 mm Hg), sol alc, other organics, diff sol w. Prepd by heating in a closed tube at 125-140° 33% aq ethylamine with propyl chloride
Refs: 1)Beil 4, 138, (361) & [621] 2) E. Comanducci & M.Arena, ChemZentr **1907** II, 1396

N-Nitro Ethylpropylamine,
$H_3C.CH_2.N(NO_2).CH_2.CH_2.CH_3$; mw 132.16, N21.20%;, col liq, sp gr 1.028 at 15°, bp 108° (22 mm Hg), Prepd from propyl iodide and the potassium salt of ethylnitramine
Refs: 1)Beil 4, 146 2)H.Umbgrove & A.P.N. Franchimont, Rec 17, 274 (1878)

Ethyl-2,2-dinitropropylamine Nitrate,
$H_3C.CH_2.NH.CH_2.C(NO_2)_2.CH_3.HNO_3$; mw 240.17, N23.33%, OB to CO_2-60%; crysts, mp 111-112°. Prepd by condensing ethylamine with 2,2-dinitropropanol to give the free base (which tends to revert to starting material), followed by addition of 70% HNO_3 in eth
Refs: 1)Beil, not found 2)H.E.Ungnade & L. W. Kissinger, JOC 30 (2), 357 (1965) & CA 62, 9043 (1965)

N-Nitro Ethyl-2,2-dinitropropylamine,
$H_3C.CH_2.N(NO_2).CH_2C(NO_2)_2.CH_3$; mw 222.16, N25.22%, OB to CO_2-64.9%; crysts, mp 82-3°. Prepd by treating the preceding compd with 100% HNO_3 in Ac_2O at 0-54°
Refs: 1)Beil, not found 2)H.E.Ungnade & L.W.Kissinger, JOC 30(2), 357 (1965) & CA 62, 9043 (1965)

2,2,2-Trinitroethyl-3,3,3-trinitropropylamine,
$C(NO_2)_3.CH_2.NH.CH_2.CH_2.C(NO_2)_3$; mw 357.15, N27.45%, OB to CO_2-6.7%, crysts, mp 64-5°(chlf). Prepd from trinitroethanol and trinitropropylamine hydrochloride in aq base
Refs: 1)Beil, not found 2)M.B.Frankel & K.Klager, JChemEngData 7, 412-13(1962) & CA 57, 10993 (1962)

N-(2,2,2-Trinitroethyl)-N-(3,3,3-trinitropropyl)-nitramine or Heptanitropropylamine,
$(O_2N)_3C.CH_2.N(NO_2).CH_2.CH_2.C(NO_2)_3$; mw 402.15, N27.86%, OB to CO_2+ 4.0%; crysts, sp gr 1.83, mp 144-145°, bp - ignites at 190° (by hot bar method). Prepd by treating the preceding compd with 100% HNO_3 and Ac_2O at 0° ambient temp. Impact sensitivity is comparable to that of PETN
Refs: 1)Beil, not found 2)L.T.Carleton & M.B.Frankel, Aerojet General Corp Rept No 660 (1952), Explosives Research Contract No 7-onr-46208 3)D.V.Sickman & W.F.Sagger, NAVORD Rept 486 (1954), Res & Devel in New ChemHighExpls 4)M.B.Frankel & K.Klager, JChemEngData 7, 412-13 (1962) & CA 57, 10993 (1962)

Ethyl-iso-propylaniline and Derivatives

N-Ethyl-iso-propylaniline, $C_6H_5N(C_2H_5)(i\text{-}C_3H_7)$; mw 163.25, N8.58%; liq, sp gr 0.9343 at 15°, bp 214-15°. Prepd from ethylaniline and i-propyl iodide

Refs: 1)Beil **12**, 167 2)J.V.Braun, Ber **33**, 2732 (1900)

N-Ethyl-(2-nitro-iso-propyl)aniline, Hydrochloride, $C_6H_5N(C_2H_5)CH(CH_3)CH_2NO_2.HCl$; mw 244.73, crysts, mp 123°. Prepd from 1-nitropropene and ethylaniline and isolated as the HCl salt

Refs: 1)Beil, not found 2)R.L.Heath & J.D.Rose, JChemSoc **1947**, 1486-9 & CA **42**, 4911 (1948)

2,4,6-Trinitro-N-ethyl-iso-propylaniline or Picryl Ethylisopropylamine, $C_6H_2(NO_2)_3N(C_2H_5)(i\text{-}C_3H_7)$; mw 298.26, N18.79%; red, ndls or yel plates (from alc or alc/HAc), heating the red at 90° converts it to the yellow which has mp 108-9° (isomerism probably due to restricted rotation of the aromatic C-N bond). Prepd by heating picryl chloride with ethylisopropylamine in alc

Refs: 1)Beil **12**, 764 2)A.Mulder, Rec **25**, 114 (1905)

Ethyl-N-propylaniline and Derivatives,

N-Ethyl-n-propylaniline, $C_6H_5N(C_2H_5)(n\text{-}C_3H_7)$; mw 163.25, N8.58%; liq, bp 107° (14 mm Hg), 216°. Prepd from ethylaniline and propylbromide

Refs: 1)Beil **12**, 167, (159) & [94] 2) A. Claus & H.Hirzel, Ber **19**, 2787 (1886)

N-Ethyl-(2-nitropropyl)aniline Hydrochloride, $C_6H_5N(C_2H_5)CH_2CH(NO_2)CH_3.HCl$; mw 244.73, mp 126°. Prepd from 2-nitropropane and ethylaniline

Refs: 1)Beil, not found 2)R.L.Heath & J.D. Rose, JChemSoc **1947**, 1486-9 & CA **42**, 4911 (1948)

2,4-Dinitro-N-ethylpropylaniline, $C_6H_3(NO_2)_2N(C_2H_5)(n\text{-}C_3H_7)$; mw 253.26, N16.59%; or-yel plates (alc), mp 55°, sp gr 1.343 at 10°. Prepd by refluxing in alc a mixture of ethylpropylamine and 2,4-dinitrobromoaniline

Refs: 1)Beil **12**, 750 2)A.Mulder, Rec **25**, 108 (1905)

2,6-Dinitro-N-ethylpropylaniline, $C_6H_3(NO_2)_2N(C_2H_5)(n\text{-}C_3H_7)$; mw 253.26, N16.59%; crysts, mp 38-40°. Prepd by reacting ethylpropylaniline with 2,6-dinitrochlorobenzene

Refs: 1)Beil, not found 2)E.Lilly & Co, Brit P917253 (1963) & CA **59**, 9889 (1963)

Ethylpyridines and Derivatives

2-Ethylpyridine, $C_2H_5C_5H_4N$; mw 107.15, N13.07%; liq, sp gr 0.9371 at 17°, bp 148.7°, dif sol in w. Prepd by heating N-ethylpyridium iodide in a tube at 300°

Refs: 1)Beil **20**, 241 & [159] 2)A.Ladenburg, Ber **16**, 2059 (1883)

2-(2-Azidoethyl)pyridine, $N_3CH_2.CH_2C_5H_4N$; mw 148.17 N37.81%; liq, sp gr 1.1122 at 20/4°, bp 65° (1 mm Hg), n_D 1.5289 at 25°. Prepd by reacting HN_3 with 2-vinylpyridine in aq HAc; there is a **Picrate**, yel ndls (alc), mp 112-13°

Refs: 1)Beil, not found 2)J.H.Boyer, JACS **73**, 5248-52 (1951) & CA **47**, 490 (1953)

2-(2-Nitroethyl)pyridine, $O_2NCH_2CH_2C_5H_4N$; mw 152.15, N18.41%; crysts, mp 145° (alc). Prepd by reacting HNO_2 with 2-vinylpyridine at 60° (Ref 2). Ref 3 [prepn by $NaBH_4$ reduction of 2-(2-nitrovinyl)pyridine] states that the material is unstable in air, likewise an **HCl salt** (mp 106-8°), but that a **Picrate salt** (mp 129-30° dec) does analyze properly

Refs: 1)Beil, not found 2)F.E.Cislak & L.H.Sutherland, USP 2490672 (1949) & CA **44**, 4043 (1950) 3)A.I.Meyers & J.C.Sircar, JOC **32** (12), 4136 (1967) & CA **68**, 28896 (1968)

2-Ethyl-5-nitropyridine, $C_2H_5(NO_2)C_5H_3N$; mw 152.15, N18.41%; liq, bp 69° (0.3 mm Hg). Prepd by reacting 2-chloro-5-nitropyridine with sodium diethylinalonate in tetrahydrofuran, followed by decarboxylation by heating in dil sulfuric acid

Refs: 1)Beil, not found 2)G.H.Cooper & R.L.Rickard, JChemSoc C. (4), 772-6 (1971) & CA **74**, 125355 (1971)

3-Ethylpyridine, $C_2H_5C_5H_4N$; mw 107.15, N13.07%; liq, sp gr 0.9401 at 22.5/4°, bp 165.9°, n_D 1.5021 at 22.5°, dif sol in w; sol in alc & eth; steam distils. Prepd by distillation of glycerine with ammonium phosphate

Refs: 1)Beil, **20**, 242 & [159] 2)C.Stoehr, JPrCh [2] **43**, 155 (1891)

3-Ethyl-4-nitropyridine, $C_2H_5(NO_2)C_5H_3N$; mw 152.15, N18.41%; yel oil, bp 56-8°, (0.25 mm Hg), n_D 1.5305 at 16°. Prepd by converting 3-ethylpyridine to the N-oxide, nitrating this at 50-95° with mixed acid to give the N-oxide intermediate (yel ndls, mp 68-9°), and further treatment with PCl_3 in chlf at 0-10°

Refs: 1)Beil, not found 2)J.M.Essery & K.Schofield, JChemSoc **1960**, 4955 & CA **55**, 7413 (1961)

4-Ethylpyridine, $C_2H_5.C_5H_4N$, mw 107.15, N13.07%; liquid, sp gr 0.936 at 20°(Lange); 0.9460 at 20°(Ref 3), bp 168°, n_D 1.5018 at 20°; v sl sol in w; sol in alc & eth. Can be prepd by heating N-ethyl-pyridinium iodide in a tube at 300°. It is dangerous when heated to decompn as toxic fumes of nitrogen oxide are emitted; can react with oxidizing materials (Ref 4)

Refs: 1) Beil **20**, 243 & [159] 2)A.Ladenburg, Ber **16**, 2059 (1883) 3)CondChemDict (1961), 476-R; 8th edit (1971), not found 4) Sax, 3rd edit (1968), 762-L

4-(1,1-Dinitroethyl)-pyridine, $(NO_2)_2CH.CH_2C_5H_4N$; mw 197.15, N21.32%; crysts, mp 55-7°. Prepd by treating with 25% H_2O_2 and 93% HNO_3 a mixture of methylol-4-ethyl pyridines at 100°. There is a **Picrate salt**, mp 137°

Refs: 1) Beil, not found 2)M.V.Rubtsov et al, ZhurObshcheiKhim **25**, 2453 - 7 (1955) & CA **50**, 9401(1956)

Ethylquinolone and Derivatives

N-Ethyl-2-quinolone or N-Ethyl-α-carbostyril, C_6H_4-CH=CH-C(O)-N(C_2H_5); mw 173.22, N8-09%; ndls (pet eth), mp 53-5°, bp 316-18°; sol in usual organics. Prepd by refluxing α-carbostyril with ethyl iodide and sodium alcoholate

Refs: 1)Beil **21**, 306, (297) & [260] 2)P. Friedlaender & A. Weinberg, Ber **18**, 1530 (1885)

N-Ethyl-5-nitro-2-quinolone, $(NO_2)C_6H_3$-CH=CH-C(O)-N(C_2H_5); mw 218.22, N12.84%; crysts (alc), mp 135°, sol in alc, benz & hot w. Prepd by treating 5-nitroquinoline ethyliodate with potassium ferricyanide

Refs: 1)Beil **21**, 309 2)H.Decker, JPrCh [2] **45**, 176 (1892)

N-Ethyl-6-nitro-2-quinolone, $(NO_2)C_6H_3$-CH=CH-C(O)-N(C_2H_5); mw 218.22, N12.84%; yel ndls (alc), mp 183°, subl. Prepd by nitrating the parent, heating ethyliodide with sodium -6-nitro-2-quinolonate, or by the potassium ferricyanide oxidation as with the 5-nitro compd

Refs: 1)Beil **21**, 309 2)H.Decker, JPrCh [2] **64**, 87, 89, 96 (1901)

N-Ethyl-6-nitro-2-quinolonium Nitrate, $(NO_2)C_6H_3$-CH=CH-C(OH)=N(C_2H_5)$^+$(ONO_2)$^-$; mw 281.23, N14.94%; yel ndls, mp 183°. Prepd from the parent and nitric acid. Loses nitric acid in w or on standing in air

Refs: 1)Beil **21**, 309 2)H.Decker, JPrCh [2] **65**, 302 (1902)

N-Ethyl-7-nitro-2-quinolone, $(NO_2)C_6H_3$-CH=CH-C(O)-N(C_2H_5); mw 218.22, N14.94%; ndls (alc), mp 168-9°. Prepd by oxidation of 7-nitroquinoline ethyliodate with alkaline potassium ferricyanide

Refs: 1)Beil **21**, 309 2)H.Decker, JPrCh [2] **64**, 88 (1901)

N-Ethyl-8-nitro-2-quinolone, $(NO_2)C_6H_3$-CH=CH-C(O)-N(C_2H_5); mw 218.22, N14.94%; yel tufts (methanol, benz/pet eth), mp 96°, sol in benz. Prepd by heating sodium-8-nitro-quinolonate with ethyliodide at 150°

Refs: 1)Beil **21**, 309 2)H.Decker & A. Stavrolopoulos, JPrCh [2] **68**, 101 (1903)

N-Ethyl-5, x-dinitro-2-quinolone, $(NO_2)_2C_6H_2$-CH=CH-C(O)-N(C_2H_5); mw 263.22, N15.97%; spears (alc), mp 197°(dec). Prepd from the 5-nitro compd by treatment with fuming HNO_3

Refs: 1)Beil **21**, 310 2)H.Decker, JPrCh [2] **65**, 303 (1902)

N-Ethyl-6-x-dinitro-2-quinolone,
$(NO_2)_2C_6H_2$-CH=CH-C(O)-N(C_2H_5); mw 263.22, N15.97%; yel plates (tol), mp 216°, subl, expl at higher temp; sol in acidic and neutral liqs. Prepd from the 6-nitro compd by treatment with concd HNO_3
Refs: 1)Beil **21**, 310 2)H.Decker, JPrCh[2] **65**, 302 (1902)

N-Ethyl-5-x,x-trinitro-2-quinolone,
$(NO_2)_3C_6H$-CH=CH-C(O)-N(C_2H_5); mw 308.22, N18.18%; crysts(tol) contain tol, mp 222°(dec). Prepd by refluxing the 5-nitro compd with fuming HNO_3
Refs: 1)Beil **21**, 310 2)H.Decker, JPrCh [2]**65**, 301 (1902)

N-Ethyl-6-x,x-trinitro-2-quinolone,
$(NO_2)_3C_6H$-CH=CH-C(O)-N(C_2H_5); mw 308.22, N18.18%; yel crysts (tol), mp 224°, sol in warm NaOH; diffc sol in hot alc. Prepd from the parent of the 6-nitro compd by vigorous nitration
Refs: 1)Beil **21**, 310 2)H.Decker, JPrCh [2]**64**, 97 (1901)

N-Ethyl-7-x,x-trinitro-2-quinolone,
$(NO_2)_3C_6H$-CH=CH-C(O)-N(C_2H_5); mw 308.22, N18.18%; plates (HNO_3), ndls (tol) contain tol; mp 237°(dec), sol in NaOH. Prepd by warming the 7-nitro compd with sp gr 1.5 HNO_3 at 100°
Refs: 1)Beil **21**, 310 2)H.Decker, JPrCh [2] **64**, 99 (1901)

Ethylsalicylate and Derivatives

Ethylsalicylate, $HO.C_6H_4.COO.C_2H_5$; mw 166.18, colorless liq, sp gr 1.13 at 25/25°, bp 234°, 139°(50 mm Hg), 102°(9 mm Hg), fr p 1.3°, n_D 1.525 at 14.4°, sol in eth & alc; insol in water. Prepd by esterification of salicylic acid with alc HCl

It is used medicinally as an antiseptic, and as a solvent for NC
Refs: 1)Beil **10**, 73, (34) & [47] 2)C.Göttig, Ber **9**, 1473 (1876) 3)CondChemDict (1971), 374-L

4-Azidoethylsalicylate,
$N_3(HO)C_6H_3COOC_2H_5$; mw 207.19, N20.28%; mp 50-1°(dec). Prepd by diazotization of 4-aminoethylsalicylate followed by reaction with hydrazine
Refs: 1)Beil, not found 2)M.J.Sullivan & C.K.Banks, USP 2633470 (1953) & CA **48**, 2107 (1954)

3-Nitroethylsalicylate,
$O_2N(OH)C_6H_3COOC_2H_5$; mw 211.18; N6.63%; plates or ndls (alc), mp 44°, sl sol in cold w and alc. Prepd by esterification of 3-nitrosalicylic acid with relfuxing alc and concd H_2SO_4
Refs: 1)Beil **10**, 115 & [66] 2)E.Zacharies, JPrCh [2] **43**, 434 (1891)

4-Nitroethylsalicylate,
$O_2N(HO)C_6H_3COOC_2H_5$; mw 211.28; N6.63%; ndls (alc), mp 87°, sol in eth & benz. Prepd by esterification of 4-nitro salicylic acid by warming with alc H_2SO_4
Refs: 1)Beil **10**, (51) & [67] 2)W.Borsche, Ann **390**, 18 (1912)

5-Nitroethylsalicylate,
$O_2N(HO)C_6H_3COOC_2H_5$; mw 211.18, N6.63%; ndl (alc), mp 92-3°, sol in alc & eth; insol w. Prepd by esterification of 5-nitrosalicylic acid with alc H_2SO_4
Refs: 1)Beil **10**, 118 & [68] 2)P.Thieme, JPrCh [2] **43**, 469 (1891)

3,5-Dinotroethylsalicylate,
$(O_2N)_2(OH)C_6H_2COOC_2H_5$; mw 256.18, N 10.94%; plates (alc), mp 99-100°, sol in hot alc. Prepd by mixed acid nitration of the ester
Refs: 1)Beil **10**, 123 2)A.Cahours, AnnChim(Paris) [3] **27**, 463 (1849)

Trinitroethyl Salicylate,
$HO.C_6H_4COO.CH_2.C(NO_2)_3$; mw 301.17, N13.95%; OB to CO_2-66.7%, crysts, sp gr 1,60, mp 71°. Can be prepd by the reaction of the acid chloride of salicylic acid with Trinitroethanol (Ref 2). Its sensitivity to impact approaches that of TNT. It can be used as a castable medium for expl compns
Refs: 1)Beil, not found 2)Hercules Powder Co, Progress Report March 1949, High Explosives, Task A, Contract NOrd 9925 3)W.F. Sager & D.V.Sickman, NAVORD Rept **483** (1952), Res & Devel in NewChemHighExpls, p 26

Trinitroethyl-3,5-dinitro-salicylate, $HO.C_6H_2(NO_2)_2.COO.CH_2.C(NO_2)_3$; mw 391.17, N17.65%, OB to CO_2 -30.8%, crysts, sp gr 1.64, mp 124°; ignites at 219°. Can be prepd by the nitration of trinitro ethylsalicylate with mixed acid (Ref 2). Its impact sensitivity is about that of Comp A

Refs: 1)Beil, not found 2)Hercules Powder Co, Progress Rept March 1949, High Expls, Task A, Contract NOrd 9925 3)W.W.Sager & D.V.Sickman, NAVORD Rept **483** (1952), p 14

Ethyl Silicate or Tetraethyl Orthosilicate, $Si(OC_2H_5)_4$; mw 208.30; col, flammable liq with faint odor; sp gr 0.9356 at 20/20°, fr p -82.5° (Lange), -77° (Ref 3); bp 168.6° (Lange), 168.1° (Ref 3); fl p 125°F, vapor press 1.0 mm at 20°; viscosity 0.0179 poises at 20°; sol in alc; hydrolyzed by W. Can be prepd from silicon tetrachloride and alc. Used as a bonding agent and for many other purposes indicated in Ref 3

Refs: 1)Beil **1**, 334 2)M.Ebelmen, Ann **57**, 334 (1846) 3)CondChemDict (1961), 476-R & 477-L; 8th edit (1971), 374-L 4) Sax, 3rd edit (1968), 762

Ethyl Stearate and Derivatives

Ethyl Stearate, $C_{17}H_{35}COO.CH_2.CH_3$; mw 146.14; col crysts, sp gr 0.848 at 36°, mp 33.8°(31.1°), bp 199-201° at 10 mm; insol in w; sol in alc & eth. Can be prepd by esterification in 3% alc HCl

Refs: 1)Beil **2**, 379, (172), [352] & {1013} 2)Holzmann, ArchPharm **236**, 440 (1898)

Trinitroethyl Stearate, $C_{17}H_{35}.COO.CH_2C(NO_2)_3$, mw 447.53, N9.39%; crysts, sp gr 1.20, mp 46°. Can be prepd by interaction of acid chloride of stearic acid with Trinitroethanol. It is an expl insensitive to impact above 160 cm on ERL machine and unstable to heat. The vacuum stability test 100° gave over 30 ml of gas per 1.0g in 48 hours

Refs: 1)Beil, not found 2)W. F.Sager & D.V.Sickman, NAVORD Rept **483** (1952), Res & Devel in New Chemical High Expls, p 35

Ethyl-substituted Acid Amides, N-Trinitro Derivatives. Their prepn was described by Th.Quadflieg, DAS 1102135, Kl 12-0-16 and abstracted in Explosivstoffe **1962**, 81-2

Ethylsuccinic Acid and Derivatives

Ethylsuccinic Acid, $HOOC.CH(C_2H_5).CH_2.COOH$; mw 146.14; col crysts, mp 98°, v sol in w, alc or eth. Can be prepd by sodium amalgam reduction of $CH_3.CH{=}C(CO_2H).CH_2.CO_2H$ in weak acid

Refs: 1) Beil **2**, 660, (279) & [584] 2) R.Fittig & I .Fränkel, Ann **255**, 41 (1889)

Ethylsuccinyl Diazide, $N_3OC.CH(C_2H_5).CH_2.CON_3$; mw 196.17, N42.83%; brownish yel oil; mp-puffs off on heating. Was prepd by Curtius by treating ethylsuccinyl dihydrazide with HNO_2 in eth

Refs: 1)Beil, not found 2)Th.Curtius, JPraktChem **125**, 82 (1930) & CA **24**, 3231 (1930)

Ethylsuccinyl Hydrazide, $H_2N.NH.OC.CH(C_2H_5).CH_2CONH.NH_2$; mw 174.20, N15.72%; ndls (alc), mp 163°, sol in alc & w. Prepd from the diethylester of the parent and hydrazine in refluxing alc. Forms a **Dipicrate salt**, yel ndls, mp 190°

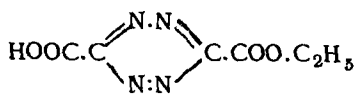
Refs: 1)Beil, not found 2)Th.Curtius, JPraktChem **125**, 79 (1930) & CA **24**, 3231 (1930)

Ethylsulfhydrate. See Ethylmercaptan in this vol

Ethylsulfuric Acid (Ethyl Hydrogen Sulfate), $C_2H_5O.SO_2.OH$, mw 126.13; col oily liq, sp gr 1.316 at 17°, bp 280° with decompn; sol in w, alc or eth; decomp by hot w or hot alc. Can be prepd by the action of sulfuric acid on ethanol. Used in medicine, org prepns and as a precipitant for casein

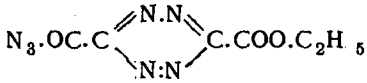
Refs: 1)Beil **1**, 325 2)CondChemDict (1961), 477-L; 8th edit (1971), 374-R 3)Sax, 3rd edit (1968), 763-L

Ethyltetrazinedicarboxylic Acid and Derivatives
Ethyltetrazinedicarboxylic Acid,



mw 198.14, N 28.28%. This compound is not found in the open literature
Refs: 1) Beil, not found 2) CA, not found

Ethyltetrazinedicarboxylic Azide (Tetrazindicarbonsäure-äthylesterazid, in Ger),



mw 223.15, N 43.94%, violet-red plates, mp – explodes on heating, sol in acet & chlf. Prepd from the corresponding hydrazide and HNO_2 in cold chlf
Refs: 1) Beil **26**, 571 2) Th. Curtius & E. Rimele, Ber **41**, 3112–13 (1908) & CA **3**, 66 (1909)

Ethyltetrazole and Derivatives
Ethyltetrazoles. Their parent compds known as **tetrazoles** can be either

1H(or α)-tetrazole, $\underset{1}{NH}\text{–}\underset{2}{N}\text{=}\underset{3}{N}\text{–}\underset{4}{N}\text{=}\underset{5}{CH}$, or

2H(or β)-tetrazole, $\underset{5}{CH}\text{=}\underset{1}{N}\text{–}\underset{2}{NH}\text{–}\underset{3}{N}\text{=}\underset{4}{N}$,

and ethyl group can be placed in position 1 of 1H-tetrazole, position 2 of 2H-tetrazole and position 5 of 1H- or 2H-tetrazoles (See Vol 1 of Encycl, p V)

The following ethyltetrazoles, $C_3H_6N_4$, mw 98.11, N 57.12, are described in the literature:

1-Ethyltetrazole, $N(C_2H_5)\text{–}N\text{=}N\text{–}N\text{=}CH$;

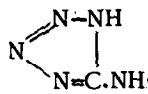
mw 98.11, N 57.12%; liq, bp 155–6° (14mm Hg), sol in alc, eth, benz & w. Prepd by heating hydrazoic acid and ethylisocyanide in eth
Refs: 1) Beil **26**, (108) 2) E. Oliveri-Mandalà & B. Alagna, Gazz **40** II, 442 (1910)

2-Ethyltetrazole, $CH\text{=}N\text{–}N(C_2H_5)\text{–}N\text{=}N$;

mw 98.11, N 57.12%; liq, bp 152–5°, 70–1° (35mm Hg). Prepd by heating 2-ethyltetrazole-5-carbonic acid at 130–40°
Refs: 1) Beil **26**, 108 2) E. Oliveri–Mandalà & T. Passalacqua, Gazz **41** II, 435 (1911)

Ethyltetrazylazide, $C_3H_5N_7$; mw 139.12, N 70.49%; crysts. Prepd by reacting ethyl chloride or sulfate with an alkali salt of tetrazylazide. No props or structure are given; presumably it is the 1-ethyl-5-azido-1H-tetrazole, $N(C_2H_5)\text{–}N\text{=}N\text{–}N\text{=}CN_3$
Refs: 1) Beil, not found 2) W. Friederich, USP 2170943 (1939) & CA **34**, 265 (1940) 3) Ibid, BritP 510992 (1939) & CA **34**, 5664 (1940)

Ethyltetrazolylcarbamate,



mw 157.14, N 44.57%; solid (from 95% alc), mp – dec 255–56°. It was prepd by refluxing 5-aminotetrazole (0.1 mol) & ethyl chloroformate (0.2 mol) for 4 hrs. This compd can be nitrated, followed by hydrolysis of the nitrocarbamate to convert it to Nitraminotetrazole. This compd is sl sol in hot w & alc; v sl sol in eth. It forms a **Silver salt**
Refs: 1) Beil **26**, [244] 2) R. Stollé, Ber **62**, 1121 (1929) 3) ADL, Synthesis HE's, 3rd Rept (1953), 305

Ethyltetryl. See N-Ethyl-(2,4,6-trinitrophenyl)-nitramine under N-ETHYLPHENYLAMINES AND DERIVATIVES in this Vol

Ethyltoluenes and Derivatives
Ethyltoluenes or Ethylmethylbenzenes, $C_2H_5.C_6H_4.CH_3$; mw 120.19. Three isomers are known: ortho-, meta- and para-. Their preps and properties are given in Refs
Refs: 1) Beil **5**, 396, (192) & [308] (ortho) 2) Beil **5**, 396, (192) & [309] (meta) 3) Beil **5**, 397, (193) & [310] (para)

3-Nitro-2-ethyltoluene, $C_6H_3(CH_3)(C_2H_5)NO_2$; mw 165.19, N 8.48%; liq, bp 75–9° (1.2mm Hg). Prepd by mixed acid nitration of the parent at 0–5°
Refs: 1) Beil, not found 2) M. Dolinsky et al, JAssocOfficAgrChemists **42**, 709–20 (1959) & CA **54**, 4268 (1960) (Also infrared spectrum)

4-Nitro-2-ethyltoluene,
$C_6H_3(CH_3)(C_2H_5)NO_2$; mw 165.19, N 8.48%; crysts, mp 23°, bp 271°, 130–31°(10mm Hg), 89–92°(1.2mm Hg). Prepd from 5-amino-2-ethyltoluene by nitration of the acetanilide (yel, mp 103°), return to the free amine (orn, mp 90°), and finally deamination with HNO_2
Refs: 1) Beil, not found 2) G.T. Morgan & A.E.J. Pette, JChemSoc **1934**, 418–22 & CA **28**, 4396–7 (1934). See Ref 2 of preceding entry for infrared spectrum

5-Nitro-2-ethyltoluene,
$C_6H_3(CH_3)(C_2H_5)NO_2$; mw 165.19, N 8.48%; liq, bp 89–92°(1.2mm Hg). Prepd by mixed acid nitration of the parent at 0–5°
Refs: 1) Beil, not found 2) M. Dolinsky et al, JAssocOfficAgrChemists **42**, 709–20 (1959) & CA **54**, 4268 (1960) (Also infrared spectrum)

6-Nitro-2-ethyltoluene,
$C_6H_3(CH_3)(C_2H_5)NO_2$; mw 165.19, N 8.48%; liq, bp 78–80°(1.2mm Hg). Prepd by mixed acid nitration of the parent at 0–5°
Refs: 1) Beil, not found 2) M. Dolinsky et al, JAssocOfficAgrChemists **42**, 709–20 (1959) & CA **54**, 4268 (1960) (Also infrared spectrum)

2-Nitro-3-ethyltoluene,
$C_6H_3(CH_3)(C_2H_5)NO_2$; mw 165.19, N 8.48%; liq, bp 72–6°(1mm Hg). Prepd by mixed acid nitration of the parent at 0–5°
Refs: 1) Beil, not found 2) M. Dolinsky et al, JAssocOfficAgrChemists **42**, 709–20 (1959) & CA **54**, 4268 (1960) (Also infrared spectrum)

4-Nitro-3-ethyltoluene,
$C_6H_3(CH_3)(C_2H_5)NO_2$; mw 165.19, N 8.48%; liq, bp 253–63°, 70–71°(0.7mm Hg). Prepd by mixed acid nitration of the 6-acetanilido-3-ethyltoluene (mp 143°), return to the free amine (gold, mp 74°), and finally deamination with HNO_2
Refs: 1) Beil, not found 2) G.T. Morgan & A.E.J. Pette, JChemSoc **1934**, 418–22 & CA **28**, 4396–7 (1934). See Ref 2 of preceding entry for infrared spectrum

5-Nitro-3-ethyltoluene,
$C_6H_3(CH_3)(C_2H_5)NO_2$; mw 165.19, N 8.48%; yel liq, bp 263–64°. Prepd by nitration in acetic acid of the 6-acetanilido-3-ethyltoluene (yel, mp 142°), return to the free amine (red, mp 64°), and finally deamination with HNO_2
Refs: 1) Beil, not found 2) G.T. Morgan & A.E.J. Pette, JChemSoc **1934**, 418–22 & CA **28**, 4396–7 (1934)

6-Nitro-3-ethyltoluene,
$C_6H_3(CH_3)(C_2H_5)NO_2$; mw 165.19, N 8.48%; liq, bp 245°(dec), 70–71°(0.5mm Hg). Prepd by nitration in acetic acid of the parent, cold
Refs: 1) Beil **5**, [310] 2) A. Mailhe, CR **173**, 160–62 (1921) & CA **15**, 3985 (1921) (CA has incorrect formula); for infrared spectrum see Ref 2 for the 2-nitro-3-ethyltoluene deriv

2-Nitro-4-ethyltoluene,
$C_6H_3(CH_3)(C_2H_5)NO_2$; mw 165.19, N 8.48%; liq, bp 247–49°, 73–76°(1mm Hg). Prepd by mixed acid nitration of the parent
Refs: 1) Beil, not found 2) O.L. Brady & J.N.E. Day, JChemSoc **1934**, 114–21 & CA **28**, 1993 (1934); for infrared spectrum see Ref 2 for 2-nitro-3-ethyltoluene above

3-Nitro-4-ethyltoluene,
$C_6H_3(CH_3)(C_2H_5)NO_2$; mw 165.19, N 8.48%; liq, bp 115–20°(10mm Hg), 69–73°(1mm Hg). Prepd by hydrazine reduction at 200° of 3-nitro-4-ethylbenzaldehyde
Refs: 1) Beil, not found 2) I.J. Rinkes, Rec **64**, 205–13 (1945) & CA **40**, 4034 (1946); for infrared spectrum see Ref 2 for 2-nitro-3-ethyltoluene above

2-(1-nitroethyl) toluene,
$C_6H_4(CH_3)CH(NO_2)CH_3$; mw 165.19, N 8.48%; liq, bp 86–89°(1mm Hg), n_D 1.5208 at 20°. Prepd by reaction of the corresponding bromo compd with $NaNO_2$ at −18° in dimethylformamide containing urea and phloroglucinol
Refs: 1) Beil, not found 2) M. Fukuyama et al, JACS **92**(15), 4697 (1970) & CA **73**, 65645 (1970)

4-(1-nitromethyl) toluene,
$C_6H_4(CH_3)CH(NO_2)CH_3$; mw 165.19, N 8.48%; liq, bp 78°(0.5mm Hg), n_D 1.5288 at 20°. Prepd in the same way as the preceding entry, from the corresponding parent
Refs: See preceding entry

x,x-Dinitro-3-ethyltoluene,
$C_6H_2(CH_3)(C_2H_5)(NO_2)_2$; mw 210.19, N 13.33%; liq, steam distils. Prepd by nitration of the parent in the cold; not further characterized or identified
Refs: 1) Beil **5**, 397 2) E. Bartow & A.W. Sellards, JACS **27**, 372(1905)

x,x-Dinitro-4-ethyltoluene,
$C_6H_2(CH_3)(C_2H_5)(NO_2)_2$; mw 210.19, N 13.33%; plates (alc), mp 51–52°, sol in boiling alc. Prepd from the parent in cold fuming nitric acid; a non-cryst oil, dinitro deriv, is also obtained. Possibly the 2,3-dinitro
Refs: 1) Beil **5**, 399 2) P. Jannasch & A. Dieckmann, Ber **7**, 1514(1874)

2,3-Dinitro-4-ethyltoluene,
$C_6H_2(CH_3)(C_2H_5)(NO_2)_2$; mw 210.19, N 13.33%; crysts, mp 51.5°. Prepd by mixed acid nitration of the parent
Refs: 1) Beil, not found (but see preceding entry) 2) O.L. Brady & J.N.E. Day, JChemSoc **1934**, 114–21 & CA **28**, 1993(1934)

2,6-Dinitro-4-ethyltoluene,
$C_6H_2(CH_3)(C_2H_5)(NO_2)_2$; mw 210.19, N 13.33%; crysts, mp 60°(62.5°). Prepd by nitrous acid deamination of the 3-amino compd [yel ndls (MeOH), mp 145°]
Refs: 1) Beil, not found 2) O.L. Brady & J.M.E. Day, JChemSoc **1934**, 114–21 & CA **28**, 1993(1934)

3,5-Dinitro-4-ethyltoluene,
$C_6H_2(CH_3)(C_2H_5)(NO_2)_2$; mw 210.19, N 13.33%; yel crysts, mp 48.5°. Prepd by mixed acid nitration at –10° of 2-acetanilido-4-ethyltoluene, return to the free amine [yel-brn plates (benz), mp 186°], and nitrous acid deamination
Refs: 1) Beil, not found 2) I.J. Rinkes, Rec **64**, 205–13(1945) & CA **40**, 4034(1946)

2,4,6-Trinitro-3-ethyltoluene,
$C_6H(CH_3)(C_2H_5)(NO_2)_3$; mw 255.19, N 16.47%; plates (alc), mp 86°(90°). Prepd by treating an intermediate dinitration product of the parent with fuming nitric acid and concd sulfuric acid
Refs: 1) Beil **5**, 397 & [310] 2) E. Bartow & A.W. Sellards, JACS **27**, 372(1905)

2,3,5-Trinitro-4-ethyltoluene,
$C_6H(CH_3)(C_2H_5)(NO_2)_3$; mw 255.19, N 16.47%; crysts, mp 82°. Prepd by mixed acid nitration of the 3,5-dinitro compd (See above)
Refs: See 3,5-dinitro-4-ethyltoluene above

2,3,6-Trinitro-4-ethyltoluene,
$C_6H(CH_3)(C_2H_5)(NO_2)_3$; mw 255.19, N 16.47%; prisms (alc), mp 93–94°. Prepd by warming the parent in mixed acid
Refs: 1) Beil **5**, 399 2) E. Glinzer & R. Fittig, Ann **136**, 314(1865)

Ethyltoluene Sulfonate and Derivatives
Ethyl-p-toluene Sulfonate, $CH_3.C_6H_4.SO_3.C_2H_5$; mw 200.26; toxic unstable crysts (from alc), sp gr 1.166 at 48/4°, mp 33–34°, bp 221.3°; insol in w; sol in alc, eth & most org solvents. It can be prepd from the sulfonyl chloride and alc Used as plasticizer for cellulose acetate and as an ethylating agent (Ref 3)
Refs: 1) Beil **11**, 99, (24) & [45] 2) (?) Jaworski, Zeit für Chemie **1865**, 221
3) CondChemDict (1961), 477-R; 8th edit (1971), 375-L 4) Sax, 3rd edit (1968), 763-R

Azido Ethyl-p-toluene Sulfonate, mw 241.27, N 17.41%. No refs to any compd fitting this name are found in the open literature
Refs: 1) Beil, not found 2) CA, not found

Nitro Ethyl-p-toluene Sulfonate, mw 245.26, N 5.71%
No refs to any para compd are found in the open literature. There is a **4-nitro-ethyl-o-toluenesulfonate**, mp 85–86°, prepd by esterification of the free acid obtained by fuming nitric acid oxidation of bis(4-nitro-2-methylphenyl)disulfide (Ref 2)
Refs: 1) Beil, not found 2) P. Pfeiffer & H. Jäger, Ber **75B**, 1885–90(1942) & CA **38**, 1218(1944)

3,5-Dinitro-ethyl-p-toluene Sulfonate or *3,5-Dinitro-4-methyl-ethylbenzene Sulfonate*, $(NO_2)_2C_6H_2(CH_3).SO_3.C_2H_5$; mw 290.26, N 9.65%; crysts, mp 82–84°(tol/hex). Prepd from the sulfonyl chloride and sodium eth oxide. The ortho-isomer is also mentioned in the same Ref, mp 87–90°, prepd in the same manner

Refs: 1) Beil, not found 2) J. Urbański, BullAcadPolSci, SerSciChim **18**(10), 617–24 (1970) & CA **74**, 111708(1971)

2,2,2-Trinitroethyl-p-toluene Sulfonate, $CH_3.C_6H_4.SO_3.CH_2.C(NO_2)_3$; mw 335.26, N 12.50%, OB to CO_2 –74.0%, crysts (benz), mp 136.5–137.0°. Prepd by heating p-toluenesulfonyl chloride with trinitroethanol and a trace of pyridine
Refs: 1) Beil, not found 2) F.G. Borgardt et al, JOC **35**(12), 4238(1970) & CA **74**, 22390(1971)

N-Ethyl-p-toluenesulfonamide and Derivatives
N-Ethyl-p-toluenesulfonamide, $CH_3.C_6H_4.SO_2.NH.C_2H_5$; mw 199.27; plates (dil alc), mp 58°(63–64°). Prepd from the sulfonyl chloride and ethylamine in alkaline solution
Refs: 1) Beil **11**, 105 & [56] 2) I. Remsen & C.S. Palmer, AmChemJ **8**, 241(1886)

N-(2-azidoethyl)-p-toluenesulfonamide, $CH_3.C_6H_4.SO_2.NH.CH_2.CH_2N_3$; mw 240.29, N 23.32%, ndls (petr eth), mp 64°, sol in usual organics. Prepd from the sulfonyl chloride and 2-azidoethylamine in cold pyridine
Refs: 1) Beil **11**, (27) 2) M.O. Forster & S.H. Newman, JChemSoc **99**, 1280(1911)

3-Nitro-N-ethyl-p-toluenesulfonamide or *3-Nitro-4-methyl-N-ethyl-benzenesulfonamide,* $NO_2.C_6H_3.CH_3.SO_2.NH.C_2H_5$; mw 244.27, N 11.47%, yel prisms (alc), mp 87°. Prepd from the sulfonyl chloride and ethylamine
Refs: 1) Beil **11**, 111 2) F.D. Chattaway, JChemSoc **87**, 160(1905)

N-Nitro-N-ethyl-p-toluenesulfonamide, $CH_3.C_6H_4.SO_2.N(NO_2).C_2H_5$; mw 244.27, N 11.47%, plates (eth), mp 69°, sp gr 1.450. No prepn is given in the Ref, possibly by reacting the sulfonyl chloride with ethylnitramine
Refs: 1) Beil **11**, [67] 2) F.M. Jaeger, ProcAcadSciAmsterdam **23**, 347–62(1920) & CA **15**, 689(1921)

3,N-Dinitro-N-ethyl-p-toluenesulfonamide or **3,N-Dinitro-N-ethyl-4-methylbenzenesulfonamide,** $NO_2.C_6H_3.CH_3.SO_2.N(NO_2).C_2H_5$; mw 289.27, N 14.53%; yel plates (CCl_4), mp 76°, sp gr 1.555. No prepn is given in the Ref, possibly by nitration of ethyl-p-toluenesulfonamide
Refs: 1) Beil **11**, [69] 2) F.M. Jaeger, ProcAcadSciAmsterdam **23**, 347–62(1920) & CA **15**, 689(1921)

N-Trinitroethyl-p-toluenesulfonamide, $CH_3.C_6H_4.SO_2.NH.CH_2.C(NO_2)_3$; mw 334.27, N 16.76%; crysts, sp gr 1.68, mp 150°, ignition temp 190°. It can be prepd by heating a mixture of p-toluenesulfonamide with Trinitroethanol. Its impact sensitivity is about that of Comp B
Refs: 1) Beil, not found 2) HerculesPowderCo, ProgressRept Sept 1949, High Expls, Task A, Contract NOrd 9925 3) W.F. Sager & D.V. Sickman, NAVORD Rept **483**(1952), Res & Devel in New High Expls, p 15

Ethyltoluidines and Derivatives
N-Ethyltoluidines or *Ethylaminotoluenes,* $C_2H_5.NH.C_6H_4.CH_3$; mw 135.20. All three isomers, o-, m-, and p-, are known
Refs: 1) Beil **12**, 786, (377) & [435] – ortho; **12**, 857 & [466] – meta; **12**, 904, (414) & [492] – para 2) CondChemDict(1961), 477-R (N-Ethyl-o-toluidine)

Azido Ethyltoluidine, mw 176.21, N 31.80%. No compd corresponding to this name is known in the open literature
Refs: 1) Beil, not found 2) CA, not found

4-Nitro-2-(ethylamino) toluene, $C_2H_5.NH.C_6H_3.CH_3.NO_2$; mw 180.20, N 15.54%; red ndls (alc), mp 81–82°. Prepd by nitrating o-ethyltoluidine in cold mixed acid
Refs: 1) Beil **12**, 845 & (393) 2) W. MacCallum, JChemSoc **67**, 247(1895)

5-Nitro-2-(ethylamino)toluene, $C_2H_5.NH.C_6H_3.CH_3.NO_2$; mw 180.20, N 15.54%; yel plates (alc), mp 98°. Prepd from the free aniline with C_2H_5I, alc and NaOH
Refs: 1) Beil **12**, 847 2) A. Bernthsen, Ber **25**, 3137(1892)

4-Nitro-3-(ethylamino)toluene,
$C_2H_5.NH.C_6H_3.CH_3.NO_2$; mw 180.20, N 15.54%; yel-red ndls (dil alc), mp 60°. Prepd from 4-nitro-3-ethoxy-toluene by heating with alc ammonia at 165–70°
Refs: 1) Beil **12**, 876 2) O. Fischer & M. Rigaud, Ber **34**, 4207 (1901)

2-Nitro-4-(ethylamino)toluene,
$C_2H_5.NH.C_6H_3.CH_3.NO_2$; mw 180.20, N 15.54%; red prisms or yel ndls, mp 47–48° (50°), sol in alc & eth. Prepd by nitrating p-ethyltoluidine with mixed acid
Refs: 1) Beil **12**, 997 2) E. Noelting & T. Stricker, Ber **19**, 549 (1886)

2,4-Dinitro-3-(ethylamino)toluene,
$C_2H_5.NH.C_6H_2.CH_3.(NO_2)_2$; mw 225.20, N 18.66%; yel ndls (alc), mp 68–69°, sol in usual organics. Prepd by warming 2,3,4-trinitrotoluene with benzylidenethylamine in alc
Refs: 1) Beil **12**, [479] 2) M. Giua & R. Petronio, JPrCh [2] **110**, 306 (1925) & CA **20**, 173 (1926)

4,6-Dinitro-3-(ethylamino)toluene,
$C_2H_5.NH.C_6H_2.CH_3.(NO_2)_2$; mw 225.20, N 18.66%; yel ndls (alc), mp 125–26°, sol in usual organics. Prepd by warming 2,4,5-trinitrotoluene with benzylidenethylamine in alc
Refs: 1) Beil **12**, [480] 2) M. Giua & R. Petronio, JPrCh [2] **110**, 306 (1925) & CA **20**, 173 (1926)

3,5-Dinitro-4-(ethylamino)toluene,
$C_2H_5.NH.C_6H_2.CH_3.(NO_2)_2$; mw 225.20, N 18.66%; orn-yel ndls (alc), mp 126–126.5°, sol in benz. Prepd by further nitration of the 3-nitro compd in nitric acid below 30°
Refs: 1) Beil **12**, 1010 & (443) 2) L. Gattermann, Ber **18**, 1485 (1885)

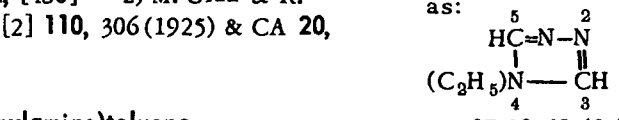
2,4,6-Trinitro-3-(ethylamino)toluene,
$C_2H_5.NH.C_6H.CH_3.(NO_2)_3$; mw 270.20, N 20.73%; yel crysts, mp 98°. Prepd from ethylamine and 2,4,6-trinitro-3-methoxy-toluene in alc
Refs: 1) Beil **12**, 879 2) J.J. Blanksma, Rec **21**, 333 (1902)

N,3,5-Trinitro-2-(ethylamino)toluene,
$C_2H_5.N(NO_2).C_6H_2.CH_3.(NO_2)_2$; mw 270.20, N 20.73%; yel crysts (alc), mp 71–72°. Prepd by refluxing diethyl-o-toluidine with fuming nitric acid
Refs: 1) Beil **12**, 852 2) P. van Romburgh, Rec **3**, 403 (1884)

N,3,5-Trinitro-4-(ethylamino)toluene,
$C_2H_5.N(NO_2).C_6H_2.CH_3.(NO_2)_2$; mw 270.20, N 20.73%; yel ndls (alc), mp 116°. Prepd by refluxing diethyl-p-toluidine with sp gr 1.48–1.52 nitric acid
Refs: 1) Beil **12**, 1012 2) P. van Romburgh, Rec **3**, 409 (1884)

Tetranitroethyltoluidine, 3-(N-Ethyl-N-nitro)-2,4,6-trinitrotoluidine or **Trinitroethylnitraminotoluene,** $C_2H_5.N(NO_2).C_6H.CH_3.(NO_2)_3$; mw 315.20, N 22.22%, OB to CO_2 –73.7%; crysts, mp 79°. Prepd by treating 2,4,6-trinitro-3-(ethylamino)toluene with sp gr 1.52 nitric acid
Refs: 1) Beil **12**, 880 2) J.J. Blanksma, Rec **21**, 333 (1902)

Ethyltriazole and Derivatives
Ethyltriazoles are **triazoles** with an ethyl group replacing one of the H's. Our way of writing the formulas of triazoles is given in Vol 1 of Encycl, p VI. Using our formulas, the compd *4-Ethyl-1,2,4-triazole* will be written as:

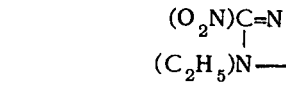


mw 97.12, N 43.25%; oil, sol in alc & w. Prepd by treating the 3-mercapto deriv with hydrogen peroxide
Refs: 1) Beil **26**, 14 2) M. Freund & H.P. Schwarz, Ber **29**, 2488 (1896)

4-Ethyl-3-nitro-1,2,4-triazole,

mw 142.12, N 39.43%. There is no open literature ref to this compd. The 5-ethyl-3-nitro compd is known, however, mp 121°. Prepd by adding the 5-ethyl-3-amino compd in 10% sulfuric acid to 10% $NaNO_2$ at 45°
Refs: 1) Beil, not found 2) L.I. Bagal et al, KhimGeterotsiklSoedin (2), 259–64 (1970) & CA **72**, 111383 (1970)

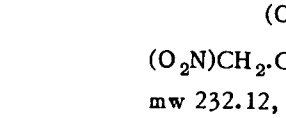
4(1)-Ethyl-3,5-dinitro-1,2,4-triazole,



mw 187.12, N 37.43%, OB to CO_2 −55.6%; solid, mp 79°. The position of ethyl group is uncertain. It was prepd by interaction of the silver salt of Dinitrotriazole and ethyl iodide. It is an expl insensitive to impact above 160cm with ERL machine, 2.5kg wt

Refs: 1) Beil, not found 2) USRubberCo, NaugatuckChemDiv, ProgressRept April-June 1949 on Contract NOrd 10121 3) W.F. Sager & D.V. Sickman, NAVORD Rept **483** (1952), p 30 4) H.P. Burchfield & D.K. Gullstrom, USP 3054800 (1962) & CA **58**, 10221 (1963)

4-(2-Nitroethyl)-3,5-dinitro-1,2,4-triazole,



mw 232.12, N 36.21%, OB to CO_2 −27.5%; crysts, sp gr 1.76, mp 147°, ignition point 274°. Prepd by treating Dinitrotriazole with Nitroethylene at RT. It is expl with impact sensitivity comparable to that of Tetryl

Refs: 1) Beil, not found 2) Univ of Louisville, Kentucky, QuarterlyProgressRept No **7** (1951), Contract NOrd 10804 3) W.F. Sager & D.V. Sickman, NAVORD Rept **483** (1952), p 7 4) D.V. Sickman, USP 2987520 (1961) & CA **56**, 4776 (1962)

Ethyltrichlorosilane, $C_2H_5.SiCl_3$; mw 163.51; colorless, flammable liquid; sp gr 1.236 at 25/25°, fr p −105.6°, bp 99.5°, n_D 1.4257 at 25°, fl p (Cleveland open cup) 57°F; readily hydrolyzed by moisture with formation of HCl. It can be prepd by reaction of ethylene and trichlorosilane in presence of a peroxide catalyst. It is used as an intermediate for silicones. This compd may form expl mixts with air. It is also highly toxic

Refs: 1) Beil **4**, 630 & (582) 2) CondChemDict (1961), 478-L; 8th edit (1971), 375-L 3) Sax, 3rd edit (1968), 763-R

Ethyl-trimethyleneglycol and Derivatives

β-Ethyl-trimethyleneglycol (2-methylolbutanol-(1), in Ger), $C_2H_5.CH.(CH_2OH)_2$; mw 104.15; liq, sp gr 0.997 at 20°, bp 86–87° (0.5mm Hg), n_D 1.4480 at 20°. Prepd by reducing ethyl diethylmalonate with copper chromite in methanol

Refs: 1) Beil, not found 2) R. Mozingo & K. Folkers, JACS **70**, 227–29 (1948) & CA **42**, 2233 (1948)

β-Nitro-β-ethyl-trimethyleneglycol, $C_2H_5.C(NO_2).(CH_2OH)_2$; mw 149.15, N 9.39%; ndls (w), mp 57–58°, sol in w, alc & eth. Prepd by reacting formaldehyde with 2-nitrobutanol-1 or with nitropropane, and potassium carbonate. Used for making polyesters

Refs: 1) Beil **1**, 483 2) J. Pauwels, ChemZentr **1898** I, 193

Note: There is no open literature reference to mono- or dinitrate derivatives of the parent or the nitro compd, but the nitro-dinitrate compd may have been prepd (mw 237.13, N 17.72%, OB to CO_2 −37.1%), presumably by nitration of the nitro compd. Possibly the lack of interest is due to the attention paid to pentaerythritol and its nitrated derivs which have similar empirical formulas

Ethyl-trimethylolmethane and Derivatives

Ethyl-trimethylolmethane or *2-Ethyl-2-Hydroxymethyl-1,3-propanediol,* $C_2H_5C(CH_2OH)_3$; mw 134.18; wh crysts, mp 57–59°, bp 160° (5mm Hg), hyg; sol in w, alc & acet. Prepd by reacting butyraldehyde and formaldehyde in the presence of alkali. Used to make polyurethane resins

Refs: 1) Beil, not found 2) I.G. Farbenindustrie A-G, BritP 484619 (1938) & CA **32**, 7609 (1938) 3) K&O **1**, 595 (1963)

Azido Ethyltrimethylolmethane, $C_2H_4.N_3.C(CH_2OH)_3$, mw 175.19. There is no reference to this compd in the open literature

Refs: 1) Beil, not found 2) CA, not found

Ethyltrimethylolmethane Nitrate, $C_2H_5C(CH_2OH)_2CH_2ONO_2$; mw 179.18. There is no reference to this compd in the open literature

Refs: 1) Beil, not found 2) CA, not found

Ethyltrimethylolmethane Dinitrate, $C_2H_5C(CH_2ONO_2)_2CH_2OH$; mw 224.18, N

12.49%, (no props given in CA). Prepd from the parent, urea, 1,3-dichloropropane, 75% HNO_3 & 75% H_2SO_4 at −15°
Refs: 1) Beil, not found 2) F. Krupp, GerP 1160427 (1964) & CA **60**, 9150 (1964)

Ethyltrimethylolmethane Trinitrate, $C_2H_5C(CH_2ONO_2)_3$; mw 269.18, N 15.61%, OB to CO_2 −50.6%; crysts, mp 51°, expl 220°, sp gr 1.48. Prepd by nitration of the parent (Ref 3). Expl props are similar to those of Tetryl
Detonation velocity – 7040 m/sec in 30mm dia
Heat of combustion at C_v – 829 kcal/m
Impact sensitivity – 3.6kg.m (4kg.m for more crystalline material)
Trauzl lead block value – 115.5%
Refs: 1) Beil, not found 2) H. Henkin & R. McGill, IndEngChem **44**, 1391–5 (1952) & CA **46**, 8857 (1952) (Expln temp, also other props) 3) L. Médard, MP **35**, 113–15 (1953) & CA **49**, 6082 (1955) 4) Ibid, **36**, 87–92 (1954) & CA **50**, 6793 (1956)

Ethylurea and Derivatives
Ethylurea, $C_2H_5.NH.CO.NH_2$; mw 88.11, ndls (alc/eth), sp gr 1.213 at 18°, mp 92°, flash p >200°F; v sol in w & alc; insol in eth. Prepd from ethylisocyanate and ammonia
Refs: 1) Beil **4**, 115, (353) & [607] 2) A. Wurtz, CR **27**, 241 (1848) 3) Sax, 3rd edit (1968), 764-L

Azidoethylurea, $N_3.CH_2.CH_2.NH.CO.NH_2$; mw 129.12, N 54.29%; scales (benz), mp 59°; sol in alc & w; insol in petr eth. Prepd from 2-azidoethyl-ammonium chloride & potassium cyanate in w
Refs: 1) Beil **4**, (360) 2) M.O. Forster & S.H. Newman, JChemSoc **99**, 1281 (1911)

N-Nitro-N'-ethylurea, $C_2H_5.NH.CO.NH.NO_2$; mw 133.11, N 31.60%; hygr ndls (eth), mp 133–34°; sol in eth. Prepd by reacting the parent with ethyl nitrate in concd sulfuric acid at −5°
Refs: 1) Beil **4**, 117 2) J. Thiele & A. Lachman, Ann **288**, 285 (1895) 3) T.L. Davis & N.D. Constan, JACS **58**, 1800–3 (1936) & CA **30**, 7543 (1936) (Prepn)

N-Trinitroethylurea, $(O_2N)_3C.CH_2.NH.CO.NH_2$; mw 223.10, N 31.40%, OB to CO_2 −10.8%; crysts, mp 175–76°. It was assumed to be the product obtd on treating an aq soln of urea with Trinitroethanol and 1 drop of HCl. It burned rapidly and could be detonated with some difficulty by impact
Refs: 1) Beil, not found 2) USRubberCo, QuarterlyProgressRept No **7** (1949), 16 3) Ditto, Rept No **8** (1949), p 25 (Description of Pilot Plant Process)

Ethylurethane and Derivatives
Ethylurethane or *Ethyl-N-ethyl Carbamate,* $C_2H_5.NH.COO.C_2H_5$, mw 117.15, colorless liq, sp gr 0.981 at 20/4°, bp 175°, n_D 1.4219 at 20°, sol in w; decompd by hot alc. It can be prepd by heating ethylisocyanate with alc at 100°
Refs: 1) Beil **4**, 114, (353) & [607] 2) A. Wurtz, JahresbFortschChem **1854**, 565

N-Nitro-ethylurethane, $C_2H_5.N(NO_2).CO_2.C_2H_5$; mw 162.15, N 17.28%; liq, sp gr 1.163 at 20/20°, bp 107° (31mm Hg), n_D 1.4432 at 20°. Prepd from the parent by nitration with fuming nitric acid and Ac_2O at 0–25°
Refs: 1) Beil, not found 2) H.M. Curry & J.P. Mason, JACS **73**, 5043–46 (1951) & CA **47**, 497 (1953)

Dinitroethylurethane, $C_5H_9N_3O_6$; mw 207.15, N 20.30%. There is no reference to this compd in the open literature
Refs: 1) Beil, not found 2) CA, not found

Trinitroethylurethane, $C_2H_5.NH.CO_2.CH_2.C(NO_2)_3$; mw 252.15, N 22.22%, OB to CO_2 −38.1%; yel oil. Prepd by either reacting nitroform with $HO.CH_2.NH.CO_2.C_2H_5$ (Ref 2) or by heating ethylisocyanate with Trinitroethanol in the presence of a little ferric acetylacetonate (Ref 3)
Refs: 1) Beil – not found 2) Nitroglycerin Aktiebolaget, BritP 813477 (1959) & CA **53**, 19885 (1959) 3) M.E. Frankel, USP 2978492 (1961) & CA **56**, 11875 (1962)

N-Nitrotrinitroethylurethane, $C_2H_5.N(NO_2).CO_2.CH_2.C(NO_2)_3$; mw 297.15, N 23.57%, OB to CO_2 −18.9%; crysts (CCl_4),

mp 50–51°. Prepd by treating the preceding entry with concd nitric acid at 100°
Refs: 1) Beil, not found 2) M.B. Frankel, USP 2978485 (1961) & CA **55**, 16429 (1961)

Éthyluréthane de la furfurine. French name for N-Carboethoxyfurfurine or Urethane Furfurine, described in Vol **2** of Encycl, p C52-R

Éthyluréthane de la morpholine. French name for N-Carboethoxymorpholine or Urethane Morpholine, described in Vol **2**, p C53-L

Ethylvalerate and Derivatives

Ethyl-n-valerate, $CH_3.CH_2.CH_2.CH_2.COO.C_2H_5$; mw 130.18; liq, sp gr 0.877 at 20°, fr p –91.2°, bp 145.5°; sl sol in w, misc with alc & eth. It can be prepd by electrolysis of sodium propionate and potassium ethylsuccinate
Refs: 1) Beil **2**, 300–1, (130) & [266] 2) W.V. Miller & H. Hofer, Ber **28**, 2434 (1895)

Ethyl-iso-valerate,
$CH_3.CH(CH_3).CH_2.COO.C_2H_5$; mw 130.18; liq, sp gr 0.867 at 20/4°, fr p –93.3°, bp 135°; sl sol in w, misc with alc, eth & benz. It can be prepd by reacting isopropyliodide with sodium aceticester and distilling the product from sulfuric acid
Refs: 1) Beil **2**, 312, (136) & [275] 2) E. Frankland & B.F. Duppa, Ann **145**, 84 (1868) 3) CondChemDict (1961), 472-R & 473-L; 8th edit (1971), 370-R

2-Azido-ethyl-n-valerate,
$CH_3.CH_2.CH_2.CH(N_3).COO.C_2H_5$; mw 171.19, N 24.54%; liq, bp 65–70° (2.8mm Hg). Prepd from the 2-bromo compd and NaN_3 in aq alc
Refs: 1) Beil, not found 2) R.M. Moriarty & M. Rayman, Tetrahedron **21** (10), 2877–91 (1965) & CA **64**, 3312 (1966)

5-Azido-ethyl-n-valerate,
$N_3CH_2.CH_2.CH_2.CH_2.COO.C_2H_5$; mw 171.19, N 24.54%; liq, bp 105–07° (13mm Hg). Prepd from the 5-bromo compd and NaN_3
Refs: 1) Beil, not found 2) D.H.R. Barton & L.R. Morgan Jr, JChemSoc **1962**, 622–31 & CA **57**, 910 (1962)

2-Azido-ethyl-iso-valerate,
$CH_3.CH(CH_3).CH(N_3).COO.C_2H_5$; mw 171.19, N 24.54%; liq, sp gr 1.0181 at 25°, bp 68–68.5° (8mm Hg), n_D 1.4338 at 25°. Prepd by heating the 2-bromo compd with NaN_3 in aq alc
Refs: 1) Beil **2**, 318 & (139) 2) M.O. Forster & R. Müller, JChemSoc **95**, 198 (1909)

2-Nitro-ethyl-n-valerate,
$CH_3.CH_2.CH_2.CH(NO_2).COO.C_2H_5$; mw 175.18, N 7.94%; yel liq, sp gr 1.0551 at 18/4°, bp 51–52° (0.1mm Hg), n_D 1.4595 at 18° (1.4274 at 20°); sol in alc, eth & benz. Prepd by shaking H_2O_2 in H_2SO_4 with the 2-nitroso compd
Refs: 1) Beil **2**, (132) 2) J. Schmidt & H. Dieterle, Ann **377**, 49 (1910)

3-Nitro-ethyl-n-valerate,
$CH_3.CH_2.CH(NO_2).CH_2.COO.C_2H_5$; mw 175.18, N 7.94%; liq, bp 105–10° (15mm Hg, contains 12% of the 3-bromo parent). Prepd by heating at 60° in dimethylformamide $NaNO_2$, 3-bromo-ethyl-n-valerate & phloroglucinol
Refs: 1) Beil, not found 2) W. Biernacki & T. Urbański, BullAcadPolonSci, SerSciChim **13** (5), 353 (1965) & CA **63**, 16209 (1965)

4-Nitro-ethyl-n-valerate,
$CH_3.CH(NO_2).CH_2.CH_2.COO.C_2H_5$; mw 175.18, N 7.94%; liq, sp gr 1.0955 at 20/4°, bp 94–95° (1mm Hg, Ref 2), 68–71° (1mm Hg, Ref 3), n_D 1.4315 at 20°. Prepd from nitroethane and ethylacrylate in liq ammonia (Ref 2) or with benzyltriethylammonium hydroxide as catalyst (Ref 3)
Refs: 1) Beil, not found 2) S. Wakamatsu & K. Shimo, JOC **27**, 1609–11 (1962) & CA **57**, 2050 (1962) 3) W. Biernacki & T. Urbański, BullAcadPolonSci, SerSciChim **13** (5), 353 (1965) & CA **63**, 16209 (1965)

5-Nitro-ethyl-n-valerate,
$O_2N.CH_2.CH_2.CH_2.CH_2.COO.C_2H_5$; mw 175.18, N 7.94%; liq, sp gr 1.075 at 25°, bp 250–55° (dec), n_D 1.4344 at 25°. Prepd by heating at 85° in w $NaNO_2$ and 5-bromo-ethyladipate
Refs: 1) Beil, not found 2) W. Treibs & H Reinheckel, Ber **87**, 341–5 (1954) & CA **49**, 4526 (1955)

2-Nitro-ethyl-iso-valerate,
$CH_3.CH(CH_3).CH(NO_2).COO.C_2H_5$; mw 175.18, N 7.94%; liq, sp gr 1.0701 at 20°, bp 60° (1mm

Hg), n_D 1.4269 at 20°. Prepd by reacting $NaNO_2$, 2-bromo-ethyl-iso-valerate & phloroglucinol in dimethylsulfoxide or dimethylformamide
Refs: 1) Beil, not found 2) N. Komblum et al, JACS **79**, 2507–9(1957) & CA **51**, 13795 (1957)

4-Nitro-ethyl-iso-valerate, $O_2N.CH_2.CH(CH_3).CH_2.COO.C_2H_5$; mw 175.18, N 7.94%; liq, sp gr 1.100 at 25°, bp 113° (9mm Hg), n_D 1.435 at 25°. Prepd by heating at 60° nitromethane and ethylcrotonate
Refs: 1) Beil, not found 2) J. Colonge & J.M. Pouchol, BullSocChimFr **1962**, 596–8 & CA **57**, 4534(1962)

4,4-Dinitro-ethyl-n-valerate, $CH_3.C(NO_2)_2.CH_2.CH_2.COO.C_2H_5$; mw 220.18, N 12.73%; sol in CCl_4. The only literature reference to this compd is for a procedure to extract it from a mixture with other expls. It may have been prepd by reacting dinitroethane with ethylacrylate, in analogy with the preceding mononitro compds
Refs: 1) Beil, not found 2) W. Selig, US At Energy Comm **UCRL-7873**, 10–19(1964) & CA **64**, 9497(1966)

4,4,4-Trinitro-ethyl-iso-valerate, $(O_2N)_3C.CH(CH_3).CH_2.CO_2.C_2H_5$; mw 265.18, N 15.84%, OB to CO_2 –69.4%; liq, bp 91–91.5°(0.65mm Hg), n_D 1.4537 at 20°. Prepd by refluxing in alc nitroform and vinylacetic acid
Refs: 1) Beil, not found 2) L.T. Eremenko & V.I. Grigos, IzvAkadNaukSSSR, SerKhim **1967**(6), 1351–3 & CA **67**, 108118(1967)

Tetranitro-ethylvalerate, mw 310.18, N 18.07%, OB to CO_2 –46.5%. There is no reference to this compd in the open literature
Refs: 1) Beil, not found 2) CA, not found

4,4-Dinitro-(2,2,2-trinitroethyl)-n-valerate, $CH_3.C(NO_2)_2.CH_2.CH_2.COO.CH_2.C(NO_2)_3$; mw 355.18, N 19.72%, OB to CO_2 –29.3%; crysts, mp 93.5–94.5°. Prepd by esterifying 4,4-dinitrovaleric acid with 2,2,2-trinitroethanol in the presence of polyphosphoric acid at 40–80°
Refs: 1) Beil, not found 2) D.W. Jensen, NAVORD Rept **2498**(1952) 3) M.H. Gold & K. Klager, Tetrahedron **19**, Suppl 1, 81 (1963) & CA **59**, 12627(1963) 4) J.M. Rosen et al, Microscope **19**(2), 151–6(1971) & CA **75**, 41551(1971)

Ethylvinyl Carbinol or **Pentene-1-ol-3,** $CH_2:CH.CH(OH).C_2H_5$; mw 86.13; liquid, sp gr 0.840 at 19.5/0°, bp 114.5–114.7°. Prepd from acrolein and diethylzinc
Refs: 1) Beil **1**, 443, (227) & [482] 2) G. Wagner, JRussPhysChemSoc **16**, 319(1884)

Ethylvinyl Ether or **Vinylethyl Ether,** $CH_2:CH.O.C_2H_5$; mw 72.10; colorless liq, sp gr 0.754 at 20/20°, fr p –115.3°, bp 35.5°, n_D 1.3739 at 20°, fl p <0°F; v sl sol in w; sol in alc. It can be prepd by reaction of acetylene with ethanol, followed by washing with w, drying in presence of alkali and distillation from metallic Na. It is extremely dangerous & reactive and can be polymerized in either the liquid or vapor phase. It is used as an intermediate and for copolymerization
Refs: 1) Beil **1**, 433, (224) & [473] 2) J. Wislicenus, Ann **192**, 106(1878) 3) CondChemDict (1961), 1211-R, 8th edit (1971), 928-L 4) Sax, 3rd edit (1968), 764-R

Ethylvinyl Hexoate or **Vinyl-2-ethyl Hexoate,** $CH_2:CH.OOC.CH(C_2H_5).C_4H_9$; mw 170.25; liq, sp gr 0.8751 at 20/20°, fr p –90°, bp 185.2°, fl p 165°F; insol in w. It can be prepd from acetylene, 2-ethylhexanoic acid, zinc oxide and Raney nickel heated under pressure. Used in polymers and emulsifying paints
Refs: 1) Beil, not found 2) W. Reppe et al, Ann **601**, 81–4(1956) & CA **51**, 9580(1957) 3) CondChemDict (1961), 1211-R; 8th edit (1971), 928-L 4) Sax (1968), 1229-L

Ethylvinylhexyl Ether or **Vinyl-2-ethylhexyl Ether,** $CH_2:CH.O.CH_2.CH(C_2H_5).C_4H_9$; mw 156.27; liq, sp gr 0.8102 at 20/20°, fr p –100°, bp 177.7°, fl p 135°F; insol in w. It can be prepd by reacting vinyl alcohol with 2-ethylhexanol over alkaline diatomaceous earth at 275°. Used as intermediate in

pharmaceuticals, insecticides and adhesives
Refs: 1) Beil, not found 2) R.I. Hoaglin & D.H. Hirsh, USP 2566415 (1951) 3) Cond-ChemDict (1961), 1211-R; 8th edit (1971), 928-L 4) Sax (1968), 1229-L

Ethylvinylpyridine or **2-Vinyl-5-ethylpyridine,** $(CH_2{:}CH).C_5H_3N(C_2H_5)$, mw 133.19, N 10.52%; liquid, sp gr 0.9449 at 20/20°, fr p −50.9°, bp 138° at 100mm, fl p (Cleveland open cup) 200°F; vapor pressure 0.2mm at 20°; sol in w, alc, eth & chlf. It can be prepd by heating 5-ethyl-2-[hydroxyethyl]-pyridine with concd HCl at 165°. Used as copolymer and in synthesis
Refs: 1) Beil **20**, 262 & [173] 2) G. Prausnitz, Ber **25**, 2394 (1892) 3) Cond-ChemDict (1961), 1211-R; 8th edit (1971), 928-L 4) Sax (1968), 1229-R

Ethylxylenes and Derivatives
Ethylxylenes, Ethyldimethylbenzenes or *Dimethylethylbenzenes,* $C_2H_5.C_6H_3(CH_3)_2$; mw 134.21. The following isomers are listed in refs:
4-Ethyl-o-xylene, colorless liq, sp gr 0.875 at 20/4°, fr p −67.0°, bp 189.8°, insol in w; sol in alc or eth. Prepn & other props in Ref 1
2-Ethyl-m-xylene, liq, bp 80–83° (24mm), n_D 1.5040 at 24°. Prepn in Ref 5
5-Ethyl-m-xylene, colorless liq, sp gr 0.866 at 20/4°, fr p −84.2°, bp 183.8°; insol in w; sol in alc or eth. Prepn & other props in Ref 2
4-Ethyl-m-xylene, colorless liq, sp gr 0.876 at 20/4°, fr p −63.0°, bp 188.4°; insol in w; sol in alc or eth. Prepn & other props in Ref 3
2-Ethyl-p-xylene, colorless liq, sp gr 0.877 at 20/4°, fr p −53.7°, bp 186.9°; insol in w; sol in alc or eth. Prepn & other props in Ref 4
Refs: 1) Beil **5**, 427 & [328] 2) Beil **5**, 429, (206) & [328] 3) Beil **5**, 428 & [328] 4) Beil **5**, 428, (206) & [328] 5) L.I. Smith & M.A. Kiess, JACS **61**, 995 (1939)

Azidoethylxylene, mw 175.22, N 24.00%. There are no literature references to a compd of this type
Refs: 1) Beil, not found 2) CA, not found

5-Nitro-4-ethyl-m-xylene, $C_2H_5.C_6H_2.(CH_3)_2.NO_2$; mw 179.21, N 7.82%. Infrared spectrum reported in Ref 2; the authors stated the prepn of this new compd was unimportant, and failed to list any props
Refs: 1) Beil, not found 2) A. van Veen et al, Rec **76**, 801–9 (1957) & CA **52**, 4321 (1958)

6-Nitro-4-ethyl-m-xylene, $C_2H_5.C_6H_2.(CH_3)_2.NO_2$; mw 179.21, N 7.82%; liq, bp 270–2° (sl dec). Prepd from the parent in cold concd nitric acid
Refs: 1) Beil **5**, 429 2) A. Töhl & A. Geyger, Ber **25**, 1535 (1892)

Dinitroethylxylene, mw 224.21, N 12.50%. There are no references to a compd of this type in the open literature
Refs: 1) Beil, not found 2) CA, not found

3,5,6-Trinitro-4-Ethyl-o-xylene, $C_2H_5.C_6(NO_2)_3(CH_3)_2$; mw 269.215, N 15.61%; ndls (alc), mp 121°. Prepd by nitration of the parent with mixed acid (Ref 3)
Refs: 1) Beil **5**, 428 & [328] 2) J. Stahl, Ber **23**, 992 (1890) 3) K. v.Auwers & K. Ziegler, Ann **425**, 258 (1921)

4,5,6-Trinitro-2-ethyl-m-xylene, $C_2H_5.C_6(NO_2)_3(CH_3)_2$; mw 269.215, N 15.61%; cryst, mp 181°. Prepd by refluxing the parent in mixed acid
Refs: 1) Beil, not found 2) L.I. Smith & M.A. Kiess, JACS **61**, 989–96 (1939) & CA **33**, 5373 (1939)

2,5,6-Trinitro-4-ethyl-m-xylene, $C_2H_5.C_6(NO_2)_3(CH_2)_2$; mw 269.215, N 15.61%; ndls, mp 127°, sol in alc. Prepd by mixed acid nitration of the parent
Refs: 1) Beil **5**, 429 2) R. Fittig & T. Ernst, Ann **139**, 193 (1866)

2,4,6-Trinitro-5-ethyl-m-xylene, $C_2H_5.C_6(NO_2)_3(CH_3)_2$; mw 269.215, N 15.61%; ndls (alc), mp 238°. Prepd by mixed acid nitration of the parent
Refs: 1) Beil **5**, 429 2) O. Jacobsen, Ber **7**, 1434 (1874)

3,5,6-Trinitro-2-ethyl-p-xylene,
$C_2H_5.C_6(NO_2)_3(CH_3)_2$; mw 269.215, N 15.61%; prisms (alc), mp 129°. Prepd by nitration of the parent
Refs: 1) Beil **5**, 428 2) O. Jacobsen, Ber **19**, 2516(1886)

Ethylxylidines and Derivatives
Ethylxylidines, Ethylaminoxylenes, Dimethyl-N-ethyl-anilines or *N-Ethyl-dimethylanilines,* $C_2H_5.NH.C_6H_3.(CH_3)_2$; mw 149.24. Prepns for the following isomers are given in the indicated refs:
3-(N-ethyl)-m-xylidine or *sym-N-Ethyl-m-xylidine,* liq, bp 120–22°(15–16mm Hg); **Perchlorate,** mp 171–73°; **Picrate,** mp 148–49° (Ref 2)

3-(N-ethyl)-o-xylidine or *N-Ethyl-vic-o-xylidine,* liq, bp 227–28°(Ref 1)

N-Ethyl-p-xylidine, liq, bp 222–23°(748mm Hg) (Ref 3)
Refs: 1) Beil **12**, 1101 2) Tetrahedron, Suppl **7**, 9–25(1966) 3) Beil **12**, 1137

Azidoethylxylidine, mw 190.25, N 29.47%. There are no references to this compd in the open literature
Refs: 1) Beil, not found 2) CA, not found

5-Nitro-4-(N-ethyl)-o-xylidine,
$C_2H_5.NH.C_6H_2.(CH_3)_2.NO_2$; mw 194.24, N 14.43%; orn crysts (alc), mp 71–74°. Prepd by making the p-toluenesulfonanilide of the non-ethylated xylidine, reacting with ethyl-iodide to make the N-ethyl compd, and then heating in sulfuric acid
Refs: 1) Beil, not found 2) J.J. Ursprung, USP 3179669(1965) & CA **63**, 11573(1965)

4,6-Dinitro-3-(N-ethyl)-o-xylidine,
$C_2H_5.NH.C_6H.(CH_3)_2.(NO_2)_2$; mw 239.24, N 17.57%; orn ndls (alc), mp 75–76°. Prepd from 3,4,6-trinitro-o-xylene and ethylamine in alc
Refs: 1) Beil **12**, (479) 2) A.W. Crossley & W.R. Pratt, JChemSoc **103**, 987(1913)

3,5-Dinitro-4-(N-ethyl) -o-xylidine,
$C_2H_5.NH.C_6H.(CH_3)_2.(NO_2)_2$; mw 239.24, N 17.57%; orn ndls (alc), mp 115°; sol in benz & chlf. Prepd from 3,4,5-trinitro-o-xylene and ethylamine in alc
Refs: 1) Beil **12**, (481) 2) A.W. Crossley & W.R. Pratt, JChemSoc **103**, 986(1913)

3,5-Dinitro-2-(N-ethyl)-p-xylidine,
$C_2H_5.NH.C_6H.(CH_3)_2.(NO_2)_2$; mw 239.24, N 17.57%; crysts, mp 133°. Prepd by heating trinitro-p-xylene with ethylamine
Refs: 1) Beil **12**, 1141 & [618] 2) J.J. Blanksma, Rec **24**, 51(1904)

2,4,6-Trinitro-5-(N-ethyl)-m-xylidine,
$C_2H_5.NH.C_6.(CH_3)_2.(NO_2)_3$; mw 284.24, N 19.72%; crysts, mp 122°. Prepd from 2,4,6-trinitro-5-methoxy-m-xylene and ethylamine in alc
Refs: 1) Beil **12**, 1133 2) J.J. Blanksma, Rec **21**, 331(1902)

N,3,5-Trinitro-2-(N-ethyl)-p-xylidine,
$C_2H_5.N(NO_2).C_6H.(CH_3)_2.(NO_2)_2$; mw 284.24, N 19.72%; crysts (alc), mp 36°. Prepd from 3,5-dinitro-2-(N-ethyl)-p-xylene and nitric acid
Refs: 1) Beil **12**, 1141 2) J.J. Blanksma, Rec **24**, 51(1904)

N,2,4,6-Tetranitro-5-(N-ethyl)-m-xylidine,
$C_2H_5.N(NO_2).C_6.(CH_3)_2.(NO_2)_3$; mw 329.24, N 21.27%; crysts, mp 85°. Prepd from the 2,4,6-trinitro compd above by nitration with sp gr 1.52 nitric acid
Refs: 1) Beil **12**, 1134 2) J.J. Blanksma, Rec **21**, 331(1902)

Ethylxyloside and Derivatives
Ethylxyloside, $C_2H_5.C_4H_8O_4.CHO$, mw 178.19. There is no reference to this compd in the open literature
Refs: 1) Beil, not found 2) CA, not found

Ethylxyloside Trinitrate,
$C_2H_5.C_4H_5N_3O_{10}.CHO$; mw 313.18, N 13.42%, OB to CO_2 –43.5%; crysts, sp gr 1.53 at 20°, mp 95.5°. Prepd by nitrating in HAc/Ac_2O at 0° ethylxylopyranoside; attempted purification of this cyclic ether caused it to open up to give the xyloside. Loses 3% of weight in 20 hours at 100°
Refs: 1) Beil, not found 2) G. Fleury et al, MP **31**, 107–20(1949) & CA **46**, 11686(1952)

Ethyne. Same as Acetylene described in Vol 1 of Encycl, pp A58-R to A64-R

Ethynylation. Condensation of acetylene with a reagent such as aldehyde to yield an acetylenic derivative. The best example is the union of formaldehyde and acetylene to produce butynediol, $HO.CH_2.C{:}C.CH_2.OH$
Ref: CondChemDict (1961), 478-R; 8th edit (1971), 375-R

Etincelle d'allumage. Fr for Igniting Spark

Étirage des poudres. Fr operation corresponding to Amer "extrusion of propellants". See under Filage des poudres

Etna or Hecla Explosive of Atlas Powder Co. Dynamite contg NG 40, Na nitrate 45, woodmeal 14 & Mg carbonate 1%
Ref: Pérez Ara (1945), 332

Etna Powder. An Amer Dynamite which contained NG 15 to 65 with a dope consisting of Na nitrate mixed with woodpulp and/or roasted flour
Refs: 1) Daniel (1902), 286 2) Fedoroff & Clift 4(1946), 32

Et-NENA. Designation of 1-(Ethyl)-nitramino-2-ethanol Nitrate, also known as N-(β-Nitroxyethyl)-ethylnitramine. See under Ethylaminoethanol (Ethylethanolamine and Derivatives) in this Vol, p E203-R

Etnite. Mixture of Asphaline (described in Vol 1 of Encycl, p A496-L) 92 and antimony sulfide 8%
Refs: 1) Daniel (1902), 286 2) Fedoroff & Clift 4(1946), 32

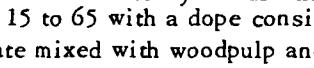
Étoupille. Fr for Primer

Étoupille à friction. Fr for Friction Primer

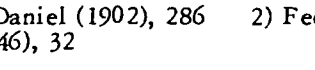
Étoupille à percussion. Fr for Percussion Primer

Euler, Leonhard (1707–1783). Swiss mathematician who lived in St Petersburg over 30 years during the reign of Catherine the Great. He was a member of the Russian Academy of Science and one of the first to study, theoretically, the processes taking place in the gun barrel during firing
Refs: 1) C.L. Barnhart, Edit, "The American College Dictionary", Random House, NY (1952), 414 2) M.E. Serebriakoff, "Vnutrenniaiya Ballistika Stvol'nykh System i Porokhovykh Raket" (Interior Ballistics of Gun Barrel Systems and of Solid Propellant Rockets), Oboronghiz, Moskva (1962). Abbreviated translation by Dr V. Nekrassoff for the US Navy, p 22 (under Bernoulli)

Eulite (Eulyt, in Ger) or **5-Methyl-4-nitro-3-(1,1-dinitroethyl)-isoxazole,** Beil name 4-Nitro-5-methyl-3-[α,β-dinitroäthyl]-isoxazol. Called by Quilico et al: *γ-Methyl-α-isoxazole-1-dinitro-2-dinitroethane,*



or



mw 246.14, N 22.76%; OB to CO_2 −52.0%; prisms (from chlf), mp 102.8°; decompd explosively when heated above mp. It was first prepd by Baup (Ref 2) in impure state, together with Dyslyt (Dislite), $C_8H_6N_4O_6$, during study of the action of nitric acid on *citraconic acid*, as was described in Vol 3 of Encycl, p C324-L. Bassett (Ref 3) prepd eulite by heating aqueous soln of citraconic acid with nitric acid (d 1.42), but he did not establish its structure. Angeli (Ref 4), prepd eulite of mp 102°. Quilico (Ref 5) prepd eulite much later by nitrating citraconic anhydride (in lieu of citraconic acid) and he suggested two structural formulas. Quilico & Fusco (Ref 6) prepd a very explosive **Mercuric Salt** of eulite by the action of mercuric chloride. It was a red-yel gelatinous compd of the formula $C_6H_6N_4O_7.HgCl_2.HgO$

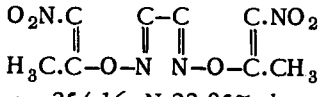
Quilico et al resumed the study of eulite in 1946 and established more definitely its

structure (Refs 7 & 8). In the same year Quilico & Fusco (Ref 9) studied the mechanism of formation of eulite

The structure of *Dislite*, $C_8H_6N_4O_6$, obtd by several investigators as a minor companion of eulite on nitration of citraconic acid was not established until 1946 (Ref 10). As Dislite was mentioned but not described in this Encycl, Vol **5**, p D1506-L, we are describing it after the refs

Refs: 1) Beil **2**, 770 & **27**, [1118] 2) S. Baup, Jahresberichte für Chemie **1851**, 405; AnnChimPhys **33**, 192(1851); Ann **81**, 102 (1852) 3) H. Bassett, ChemNews **24**, 631 (1871) & ChemZtr **43**, 157(1872) 4) A. Angeli, Ber **24**, 1303(1891) 5) A. Quilico, Gazz **65**, 1203–13(1935) & CA **30**, 5219–21 (1936) 6) A. Quilico & R. Fusco, Gazz **66**, 278–99(1936) & CA **31**, 1805(1937) 7) A. Quilico et al, Gazz **76**, 30–43(1946) & CA **41**, 382(1947) 8) A. Quilico & M. Freri, Gazz **76**, 87–107(1946) & CA **41**, 383(1947) 9) A. Quilico & R. Fusco, Gazz **76**, 195–99 (1946) & CA **41**, 385(1947) 10) R. Fusco & S. Zumin, Gazz **76**, 223–38(1946) & CA **41**, 2039(1947)

Dislite (Dyslyt, in Ger)



mw 254.16, N 22.05%; long fine ndls (from alc), mp 200.5°; sol in alc; insol in w. It is formed simultaneously with Eulite by reaction of citraconic acid & nitric acid. It forms numerous salts (Refs 1, 2 & 3)

Refs: 1) Beil **2**, 770 & {1939}; **27**, [869] {Compd called Bis[4-nitro-5-methylisoxazolyl-(3)] and α,α'-Dimethyl-β,β'-dinitro-γ,γ'-diisoxazol} 2) R. Fusco, Gazz **68**, 380–86(1938) & CA **32**, 9066(1938) 3) R. Fusco & S. Zumin, Gazz **76**, 223–38(1946) & CA **41**, 2038(1947)

"Eumuco" Shell Forging Press is a vertical type press which combines punching and drawing operations. It was designed and manufd by Eumuco AG, Leverkusen-Schlebush and used during WWII by the following Ger plants: Kronprinz AG; Immigrath, Gutehoffnungshütte AG; and Sterkrade-Kieserling & Albrecht AG, Solingen & Hasenclever AG, Düsseldorf

Refs: 1) BIOS Final Rept **668**(1946) 2) PATR **2510**(1958), p Ger 45-L

Eutectic Explosive Mixtures

Eutectic is a mixture of two or more substances which has the lowest constant melting point of any mixture of its constituents. Usually the molecular ratios of the components can be expressed in simple whole numbers, such as 1:1, 1:2, 2:3, etc

Following Table lists various eutectic mixtures of explosives. See also BINARY, TERNARY AND QUATERNARY MIXTURES in Vol **2** of Encycl, pp B116 to B120

Note 1: Accdg to Ref 2, p 1026, the binary eutectic temp 26.4° for DNT+TNT was lowered to 25.7° by addns of small quantities of TNX; similarly temp 33.8° of MNT+TNT was lowered to 30.55°, and temp 45.1° of DNT+TNT was lowered to 42.3°

Note 2: Accdg to Ref 14, AN forms the following ternary eutectics: 1) AN 66.5, Na nitrate 21.0 & K nitrate 12.5% (fr p 118.5°); and 2) AN 69, Ca nitrate 18 & Na nitrate 1.3% (fr p 107.5°)

Note 3: Eutectic temperature of ternary mixture of three TNT isomers (α 43.5, β 20.0 and γ 36.5%) was given as 44.4° by W.H. Gibson et al in JCS **121**, p 282(1922)

Refs: 1) N. Efremov, BullAcadSciPetrograd **1915**, 1309–36 and **1916**, 21–46 2) J. Bell & J. Sawyer, JIEC **11**, 1025(1919) 3) J. Bell & C. Herty, JIEC **11**, 1124–33(1919) 4) C.A. Taylor & W.H. Rinkenbach, IEC **15**, 73, 795 & 1070(1923) 5) N. Efremov & A. Tikhomirova, CA **21**, 3802(1927) 6) Ibid, CA **23**, 2349(1929) 7) Ibid, CA **23**, 3214 (1929) 8) A. Holleman, Rec **49**, 112(1930) 8a) Marshall (1932), 233 9) T. Urbański, CA **28**, 27(1934) 10) K. Hrynakowski & Z. Kapuściński, CA **28**, 6706(1934) 11) T. Urbański, CA **29**, 6129(1935) 12) T. Urbański & B. Kwiatkowski, CA **29**, 6129 (1935) 13) T. Urbański, CA **30**, 5863(1936) 14) T. Urbański & S. Kolodziejczyk, CA **30**, 5863(1936) 15) J. Timmermans, "Les Solutions Concentrées", Masson, Paris (1936), p 520 (List of several eutectic mixtures) 16) E. Burlot & P. Tavernier, MP **31**, 39(1949) [Included are curves of mp's vs compns of binary mixtures: DNCB (mp 49.5°) + PA(121°) and DNCB+TNT (79.5°). Eutectics for mixtures are: 38° for DNCB 25 & PA 75%; and 34° for DNCB 35 & TNT 65%] 17) G. Desseigne, MP **31**, 48–49(1949) (Ethyltetryl forms with 30–38% of Tetryl an eutectic of mp 75.5°)

Table
Binary Eutectic Mixtures

Components and Their Melting Points	Approximate % by Weight Ratios	Eutectic Temp °C	References
p-MNT (50.25°) – DNT (69.54°) Molecular Ratio (1:1)	45:55	26.4°	3, p1127
p-MNT (50.25°) – TNT (80.35°) Molecular Ratio 2:1	55:45	33.8°	3, p1127
DNT (69.54°) – TNT (80.35°) Molecular Ratio 1:1	55:45	45.1°	3, p1127
DNT (71°) – TNT (80.6°)	54:46	45°	8a, p233
TNT (80.6°) – 2,4,5-TNT (101.5°)	61:39	58°	8a, p233
TNT (80.6°) – Tetryl (127.5°)	52:48	65-68°	8a, p233
TNT (80.6°) – Tetryl (127.5°)	67:33	65-68°	8a, p233
TNT (78.8°) – Tetryl (126.8°)	63.4:36.6	58.8°	5, p3803
TNT (80.27°) – Tetryl (128.72°) Molecular Ratio 2:1	61.3:38.7	67.4°	4, p73
DNT (68.8°) – PA (119°)	60:40	51.6°	8a, p233
TNT (80.27°) – PA (121.8°)	69.8:30.2	59.4°	4, p795
TNT (80.6°) – PA (121.7°)	67:33	55.5°	
Tetryl (128.72°) – PA (121.8°) Molecular ratio 1:1	55.6:44.4	70°(about)	4, p1070
p-MNT (51.2°) – TNX (182.0°)	98:2	50.5°	2, p1026
DNT (69.4°) – TNX (182.0°)	94:6	67.7°	2, p1026
TNT (80.5°) – TNX (182.0°)	92:8	74.8°	2, p1026
m-DNB (69.5°) – Tetryl (126.8°)	66.5:33.5	65.5°	5, p3803
DNT (69.4°) – Tetryl (126.8°)	79.5:20.5	59.1°	5, p3803
DNP (111.4°) – Tetryl (126.8°)	43.3:57.7	83.1°	5, p3803
PA (122.4°) – Tetryl (126.8°)	43:57	75-77°	5, p3803
Styphnic Acid (175.5°) – Tetryl (126.8°)	74.5:25.5	83°	5, p3803
TNCr (101.2°) – Tetryl (126.8°)	63.5:36.5	78°	5, p3803
TNX (180.2°) – m-DNB (89.5°)	17.8:82.2	76.4°	6, p2349
TNX (180.2°) – PA (122°)	21.7:78.3	105.8°	6, p2349
TNX (180.2°) – TNCr (101.2°)	17.2:82.8	84.6°	6, p2349
TNX (180.2°) – Styphnic Acid (175.5°)	37.5:62.5	141.3°	6, p2349
TNX (180.2°) – Tetryl (126.8°)	23.5:76.5	110.8°	6, p2349
TNX (180.2°) – Picryl Chloride (83°)	13.0:87.0	73.2°	6, p2349
TNX (180.2°) – TNB (121°)	16.4:83.6	104.6°	6, p2349

(Continued)

(Continuation)

Table
Binary Eutectic Mixtures

Components and Their Melting Points	Approximate % by Weight Ratios	Eutectic Temp °C	References
Tetryl (126.8°) – Picramide (184.2°)	86.1:13.9	110.8°	7, p3214
Tetryl (126.8°) – DNAn (176°)	80.7:19.3	98.8°	7, p3214
Tetryl (126.8°) – Picryl Chloride (81.2°)	39.4:60.6	57.8°	7, p3214
Tetryl (126.8°) – TNAns (63.8°)	29.5:70.5	22.8°	7, p3214
MHeN (l 12.5°) – p-MNT (50.2°)	12.5:87.5	49.2°	9, p27
MHeN (112.5°) – m-DNB (89.5°)	52.5:47.5	65.5°	9, p27
MHeN (112.5°) – DNAns (94.5°)	55:45	77.6°	9, p27
MHeN (112.5°) – TNB (121.5°)	55:45	78.7°	9, p27
MHeN (112.5°) – TNT (80.2°)	42.5:57.5	62.8°	9, p27
MHeN (112.5°) – PETN (140°)	20:80	101.3°	9, p27
MHeN (112.5°) – ErN (61°)	18.5:81.5	57.6°	9, p27
ErN (61°) – m-DNB (89.5°)	70:30	42.4°	9, p27
ErN (61°) – p-MNT (54.5°)	47:53	32.4°	9, p27
ErN (61°) – PETN (140°)	95:5	59.5°	9, p27
PETN (140°) – p-MNT (54.5°)	10:90	50.2°	9, p27
PETN (140°) – m-DNB (89.5°)	20:80	82.4°	9, p27
PETN (140°) – DNAns (94.5°)	20:80	94.7°	9, p27
PETN (140°) – TNB (121.5°)	30:70	101.1°	9, p27
PETN (140°) – TNT (80.3°)	13:87	76.1°	9, p27
ErN (61°) – DNT (69.4°)	61:39	40.1°	13, p2834
ErN (61°) – TNB (121.5°)	67:33	45.8°	13, p2834
ErN (61°) – TNAns (68.4°)	16:84	58.6°	13, p2834
ErN (61°) –TNAns (68.4°)	76:24	52°	13, p2384
AN (210°, decomp) – $Ca(NO_3)_2$ (561°)	71:29	111°	14, p5863
HNDPhA (238°) – TNT (80.6°)	12:88	78.2°	7a, p233
Picryl Sulfide (230.5°) – TNT (80.6°)	13.5: 86.5	78.3°	7a, p233

Abbreviations: AN – Ammonium Nitrate; DNAn – Dinitroaniline; DNAns – Dinitroanisole; DNB – Dinitrobenzene; DNCB – Dinitrochlorobenzene; DNCr – Dinitrocresole; DNPh – Dinitrophenol; DNT – 2,4,-Dinitrotoluene; ErN – Erythritol Tetranitrate; HNDPhA – Hexanitrodiphenylamine; MHeN – Mannitol Hexanitrate; MNPh – Mononitrophenol; MNT – Mononitrotoluene; PA – Picric Acid; PETN – Pentaerythritol Tetranitrate; TNAn – Trinitroaniline; TNAns – Trinitroanisole; TNB – Trinitrobenzene; TNCr – Trinitrocresole; TNPh – Trinitrophenol; TNT – 2,4,6-Trinitrotoluene; TNX – Trinitro-m-xylene

Ternary Eutectic Mixtures

% by Weight

MNT	48.5	43.5	52.5	44	41	10.0	25.0	35.0
DNT	41.5	36.5	10.0	25	30	46.7	39.8	35.2
TNT	10.0	20.0	37.5	31	29	43.3	35.2	29.8
Eutectic Temp, °C	23.5°	18.95°	29.35°	20.8°	17.8°	39.2°	25.4°	17.5°

Reference 3, p1130

Eutectic Alloys. See under Fusible Alloys

Euxenite (Loranskite). (Y, Ca, Ce, U, Th)-(Nb, Ta, Ti)$_2O_6$. A rare-earth mineral, occurring in Norway, Madagascar, Canada & Pennsylvania. It is brownish-black, brilliant to vitreous; sp gr 5.0–5.9 and hardness 5–6. Used as a source of uranium, niobium and tantalum
Ref: CondChemDict (1961), 480-R; 8th edit (1971), not found

Evaluating Explosives and Hazardous Materials, Methods of US Bureau of Mines for are described by C.M. Mason & E.G. Aiken as IC (Information Circular) **8541** (1972); can be ordered from Supt of Document, US Govt Printing Office, Washington, DC 20402

Evaluation of Toxic Afterdetonation Gases Formed by Industrial Explosives. Translation of the title of paper by A.G. Streng in Explosivstoffe **19**, March/April 1971, pp 58–64. Abstracted in Expls & Pyrots **5**(8)(1972)

Evangelidi Explosive (1904). K chlorate 62.5, K ferrocyanide 30.0, charcoal 4.5 & glycerine 3.0%
Ref: Giua, Trattato **6**(I)(1959), 398

"Evanohm". Trademark of Wilbur B. Driver Company, Newark, NJ for an alloy of 75% Ni, 20% Cr, 2.5% Al & 2.5% Cu. It has been used in the form of wire in precision wound resistors
Ref: CondChemDict (1961), 480-R; (1971), p 376-R

Evaporation. See under DISTILLATION in Vol **5** of Encycl, pp D1509 to D1511 and also under Drying, pp D1560-R to D1565-L

"Everdur". Trademark of Anaconda American Brass Co, Waterbury, Conn 06720 for a group of Cu-Si alloys, with compns adjusted to hot and cold working, hot forging, welding, free machining, and for ingots for remelting and casting. The most widely used alloy in this group is "Everdur 1010", with nominal compn of Cu 95.80, Si 3.10 & Mn 1.10%. In most environments "Everdur" alloys are equivalent to Cu in corrosion resistance
Ref: CondChemDict (1961), 481-L; (1971), p 377-L

Everling & Kandler patented in 1894 (Ref 1) a smokeless proplnt contg nitrated wood cellulose previously treated to render it amorphous. For this pretreatment woodpulp was left for 10–12 hours in 10% Na hyposulfite soln maintained at 30° and then washed with water and dried. Nitration was done in the same manner as for cotton (Ref 2)
Refs: 1) FrP 238318 (1894) 2) Daniel (1902), 287

Eversoft Plastex (British). NG+NGc 25.5, CollodCotton 0.5, Di- & Tri-nitroluenes 2.5, AN 35, woodmeal 2.5 & Na chloride 34%. Ballistic Pendulum Swing 2.45 inches, vs 3.27 for 60% Gelignite (Standard)
Ref: Marshall **3**(1932), 120

Eversoft Seamex (Brit). NG+NGc 10, AN 66, woodmeal 9 & NaCl 15%. Ballistic Pendulum Swing 2.67 inches
Ref: Marshall **3**(1932), 120

Eversoft Tees Powder (Brit). NG 10, AN 60, woodmeal 10 & NaCl 20%. Ballistic Pendulum Swing 2.55 inches (See also Tees Powder)
Refs: 1) Marshall **3**(1932), 120 2) Thorpe **4**(1940), 556

EW. Abbr for Electronic Warfare

EW (Exploding Wire). See EXPLODING BRIDGE WIRE (EBW) in this Vol

EWP Thickened Fuels were described by T.L. Wheeler in OSRD **5524** (PBL 28624) (1945) and in OSRD **5524a** (PBL 28625)(1945) under the title "Thickened EWP Fuels and Ejection Devices for EWP Fuels"

The EWP (Eutectic White Phosphorus) fuel consists of white phosphorus 55 & phosphorus sesquisulfide 45% by weight. The compn by elements is 80% P & 20% S. The compn of the fuel is not critical and a reasonable deviation is allowable. The fuel is a clear, yel, heavy liq of low surface tension, moderate viscosity, and oily appearance. The specific gravity of the eutectic is 1.840 at 20° and it freezes at approx −42°C. Upon exposure to light for several days, it gradually deteriorates and becomes turbid. When stored in the dark, or in opaque containers, no deterioration takes place

When the above EWP is ejected from a nozzle it ignites and the flaming liq tends to spray out into the air in a bushy pattern somewhat resembling that obtd when using unthickened hydrocarbon fuels in a conventional portable flame thrower. It was considered desirable to modify the EWP fuel so as to obtain it in thickened, preferably gel, form for improved ballistic characteristics & increased range. The most successful mixt found was an emulsion of equal parts by wt of rubber cement & EWP, preferably with the addn of Na bicarbonate. Very good combustion and a range of 60 yds were obtd with a mixt of 8 gallons of asphalt flux & 1 gal of EWP. EWP fuels are ejected by blame-throwers or other ejection devices

EWPh. Abbr for Exploding Wire Phenomena. See W.H. Richardson, "Exploding Wire Phenomena: A Bibliography", Sandia Corp Rept **SCR 53** (Nov 1958) [Contract (AEC) AT-(29-1)-789]. This survey of the literature covers all forms of reference materials – books, periodicals & reports – published thru April 1958. It includes early material on basic physics & props of materials

Exactor. A "cartridge-actuated device" (See Vol **2** of Encycl, p C71) intended to release the safety mechanism of another cartridge-actuated device. The exactor operates by gas pressure supplied from the "initiator"
Ref: Glossary of Ord (1959), 109-R

Exaltation. The amt by which the molecular refractivity of a compd exceeds the sum of the refractivities of its atoms; it is an indication of constitution (presence of double bonds, etc)
Ref: Hackh's (1944), 328-L

Excavation, Instant (of Corps of Engineers). See Explosive Excavation, Instant

Excavation by Means of Explosives is described in Blasters'Hdb (1966), pp 385–92 (under Grade Construction) and pp 473–74 (Excavating in Hard Ground)

Excelsior Mill. It consists of two vertical discs, one of which is securely fastened to the casing of the machine, while the other is keyed to a revolving shaft. The surfaces of the discs which face each other are fitted with cylindrical teeth arranged in circles of constantly-decreasing diam as they approach the center of the plates. Teeth of the rotating disc pass between those of the fixed disc. The movable disc revolves at high speed, the material being fed at the center of the disc and delivered from the periphery in a very fine state of division

The Excelsior Mill was used for grinding of oxidizing materials & other constituents used in expl compns

Refs: 1) Colver (1918), 462 2) Perry (1963), not found

Excentric Pressed Propellant Grains. Supposedly concentric proplnt grains are usually slightly excentric. So long as an excentric grain does not have its exterior & interior burning surfaces cutting each other, it behaves like a concentric grain and the unburned or burned portions act as expected. If the proplnt grain is not excentric, the proportionality is maintained. After contact is established betwn interior & exterior burning surfaces, the burned portion increases more slowly with time than with concentric grains. Correspondingly, the unburned portion diminishes more slowly. Thus, by excentric pressing, the burning time of a proplnt may be increased

Ref: E. Kleider, SS **38**, 131–33 (1943) & CA **38**, 2823 (1944)

Exchange Coefficient. See Coefficient d'échange (Fr) in Vol **3** of Encycl, p C389-L

Exchange Reaction. A process whereby atoms of the same element in two different molecules or in two different positions in the same molecule transfer places. Exchange reactions are usually studied with the aid of a tracer or tagged atom

Ref: CondChemDict (1961), 481-L

Excitation or Activation. The disturbance of an electron so that it passes from its normal orbit to one of higher energy content as the result of the absorption of radiation. It differs from ionization in the degree to which the electron is separated, as expressed by the equations:

Excitation	*Ionization*
a) $H_2+h\nu=H_2^*$	c) $H_2=H_2^+ +e$
b) $H_2+n(h\nu)=2H^*$	d) $H_2=H^+ +H^-$
	e) $H_2=H+H^+ +e$

where $h\nu$ is a quantum, e an electron, H_2^* an excited and H_2^+ an ionized molecule, H^* an excited and H^+, H^- ionized atoms. On returning from the excited to the normal state, the absorbed radiation is emitted, ie, the substance is luminescent or fluorescent

Ref: Hackh's (1944), 328-L

Excitation. Accdg to Glossary of Ord (1959), p 109-R, it can be applications of 1) signal to the input of a vacuum tube amplifier; 2) signal power to a radio transmitting antenna; or 3) current to the transmitting or receiving elements of a synchro or self-synchronous device

Excrement (Carbonized) Explosives. The following mixtures were patented in Canada during WWI: AN 50–80, carbonized excrement 10–20, metals or metal compds 5–20 & organic nitrocompds 2–20%

Ref: J. Gronud, CanadP 165018 (1915) & CA **9**, 3363 (1915)

EXE Powder. Brit brown powder used for a while after 1876 in cannons of 152mm as a replacement for BkPdr, known as P (Pebble) Powder, which was in the form of cubes. EXE Powder was in the form of prisms with one central canal and with one of its bases being hollow and with grooves

Ref: Daniel (1902), 287 & 595

Execution of Safety. The chem & physical props of an expl determine its capabilities. As these props are "built in" it is important that expls be selected from manufacturers who exercise great care in making their products and who have the necessary research, physical & chem control, manufg skill, and test facilities to make certain that their expls measure up to the high degree of uniformity & high quality reqd by modern industry. In considering the props of expls that affect execution of safety, the following are important: strength, sensitiveness, stability, rate of deton, water resistance & consistency
Ref: J.J. Berliner & Staff, "Explosives", Pamphlet, NY (1953)

Exercise Head, Guided Missile. An item designated to simulate a warhead of guided missile. It may or may not contain telemetering devices and/or flash signals
Ref: Glossary of Ord (1959), 109-R

Exercise Head, Torpedo. An item designed for attachment to a torpedo main assemblage to complete a torpedo for a practice run. It may contain recording instruments
Ref: Glossary of Ord (1959), 109-R

Exerciser, Recoil Mechanism. An item specifically designed for exercising hydropneumatic recoil mechanism. It usually consists of a power unit, a hydraulic system and a control system. It may be trailer mounted

Recoil mechanism can also be artificially exercised by mechanical means, without removing it from storage. The machine for such exercising is known as **gymnasticator**
Ref: Glossary of Ord (1959), pp 109-R & 144-L

Exo. A prefix indicating that an element or radical is substituted for hydrogen atom attached to a side chain atom, while "eso" means substitution in the ring
Refs: 1) A. Baeyer, Ber **17**, 962(1994)
2) Hackh's (1944), p 382-R

Exograph. A radograph taken by means of X-rays (röntgenograph). A radograph taken with γ rays is known as gammagraph
Ref: Hackh's (1944), pp 328-R & 719-R

Exos (Upper-Atmosphere Research Rocket). A high-altitude sounding rocket developed for the Air Force Cambridge Research Center by University of Michigan ResInst with the assistance of NASA. Diam of rocket 22.88 inches, length 42.46 ft, payload 40 lbs and altitude 300 miles. It is a three-stage rocket consisting of an Honest John (booster), a Nike-Ajax (booster) 2nd stage and a Recruit rocket as 3rd stage. Payload consisted of a nose cone, skin temp sensing equipment and a radar beacon & telemetry devices
Ref: F.I. Ordway & R.C. Wakeford, "International Missile and Spacecraft Guide", McGraw-Hill, NY (1960), USA/63

Exothermic Reaction. A reaction in which heat is liberated. It usually proceeds rapidly; sometimes explosively. In an *endothermic* reaction a chemical change proceeds slowly with absorption of a definite number of calories
Ref: Hackh's (1944), 328-R (Exothermic); 305-R (Endothermic)

Exothermic Reaction Mixtures for Underwater Propulsion and Ignition Devices. Accdg to Lemke (Ref) of Aerojet-General Corp, pressure stability and burning rate of solid rocket fuels are improved during combstn by incorporating finely divided iron in a quantity of about 1%. The proplnts investigated were mixtures of finely divided metals: Al, Be & Mg with perchlorates of K, Li & Na
Ref: A.E. Lemke, GerP 1158884(ClC 06d), Dec 5, 1963 & CA **60**, 9094(1964)

Exotic Fuels. Accdg to Ref 1, this name is applied to *high energy fuels* (HEF) for jets and rockets which have higher performance rates than hydrocarbon fuels, such as kerosene or JP-6, etc. The term has also been used to refer specifically to those jet and rocket fuels based on boron hydrides. Such fuels, known as *Boranes*, are described in Ref 2

Exotic fuels are also known as *ultimate fuels for jets and rockets* [G.A.W. Boehm, Fortune (Dec 1957), pp 165–68, 170, 172, 177 & 178] (See also Jet Fuels and Rocket Fuels)

Refs: 1) CondChemDict (1961), pp 481-R & 575-R 2) Fedoroff & Sheffield, Encycl, PATR **2700**, Vol **2**(1962), pp B254-R to B256-R (Boron Hydride Fuels); B253-R to B254-R (Boron Hydrides)

Exotic Propellants. Like exotic fuels, exotic propellants are novel propellants that offer the promise of greater energy. Propellants that have different common linkages, such as B-H and C-F, can theoretically liberate greater energy by oxidation than can the common hydrocarbon propellants because greater net energy is available after oxidation of such linkages. Note that fluorine is the oxidizer and boron is the reducing agent in such arrangements. It is also important to remember that boron, used as a fuel alone liberates 29000 BTU/lb when oxidized compared with 19000 BTU/lb liberated by hydrocarbons; a 40% increase

Ref: Anon, "Elements of Ordnance Engineering", Part 1, Ordnance Corps Pamphlet ORDP **20–106**(1960), pp 3–26 & 3–27

Expanding Bullets. See Expansive Bullets

Expanding Jet Theory of Jones. See Detonation and Explosion Nozzle Theory of Jones in Vol 4 of Encycl, pp D460-R & D461-L & R

Expansion. An increase of dimension. It can be: *cubical or volume expansion* – increase in three dimensions; *linear* – increase in length; *thermal* – increase in volume due to increase in temperature. The rate varies with temp and depends on the *coefficient of thermal expansion*, which is defined in Ref 2 and described more fully below under "Expansion, Thermal Coefficient of"

Refs: 1) Hackh's (1944), 328-R 2) PATR **2700**, Vol 3(1966), C391-L

Expansion Equations are given in Hackh's (1944), p 328-R

Expansion, Lateral, also known as **Dispersion in Detonation** is described in Vol 4 of Encycl, pp D421-R to D423-L, under "Detonation (and Explosion), Lateral Expansion (Dispersion) and Lateral Loss in"

Expansion Ratio. In *jet propulsion* it is the ratio of the nozzle exit section area to the nozzle throat area. In *cartridge actuated device*, it is the ratio of final to initial volume in a stroking type of CAD

Ref: Glossary of Ord (1959), 109-R

Expansion, Thermal Coefficient of. Coefficient of Thermal Expansion (Wärmeausdehnungs-koeffizient, in Ger) is defined in Vol 3 of Encycl, p C391-L

The general equation which is applicable to linear, superficial or cubical coefficients of expansion, may be expressed as:

$$m_t = m_o (1 + \alpha t + \beta t^2 + \gamma t^3 + ...),$$

where: m_t = length, area or volume at t°C

m_o = same at temp 0°C

α, β, & γ = empirically detd coefficients

For solids, if a linear coeff (ℓ) is known, the superficial coeff is approx (2ℓ) and the cubical (3ℓ)

Coefficients of thermal expansion of expls and related compds are given in this Encycl, wherever available, under each compd

Refs: 1) J. Reilly & W.N. Rae, "Physico-Chemical Methods", VanNostrand, NY, Vol 1 (1943), pp 503 & 505 2) J.H. Perry, "Chemical Engineers' Handbook", McGraw-Hill, NY (1950), pp 200–202 3) P. Hidnert & W. Souder, "Thermal Expansion of Solids", USNatlBurStandards Circular No 486, USGovtPrintingOffice, Washington, DC (1950)

Expansion Wave. In aerodynamics *expansion wave* is a pressure wave that has the effect of decreasing the density of air as the air passes thru it, while the *compression wave* has the effect of increasing density of air.
Ref: Glossary of Ord (1959), p 109-R (Expansion Wave); p 78-L (Compression Wave)

Expansive or Expanding Bullets. Before about 1860, all small arms were smooth bore and bullets were ball-shaped & made of lead. Since the balls were loaded thru the muzzle (on top of previously introduced BkPdr charge serving as a propellant), it was not convenient to make them fit too tightly in the barrel. As result of this, part of propelling gases escaped during firing thru small openings betw the bullets and the barrel towards the muzzle, without contributing to propelling force. This was undesirable because it decreased the muzzle velocity and shortened the range

In order to prevent the loss of propelling gas and thus improve the performance of small arms, W. Greener and Captain Minie introduced in the 2nd quarter of the 19th century the so-called expansive or expanding bullets. They were lead bullets which were enlarged in diameter by driving into them, during firing, of speciál plugs which were placed on top of the BkPdr charge and under the bullet

Small arms using Minie's bullets were adopted by the French Army about 1849 and used as late as the Crimean War (1853–1856), although at that time some rifled weapons started to be used
Refs: 1) J.R. Newman, "The Tools of War", Doubleday, Doran, NY (1943), p 42

Expedite. One of the Brit permitted expls contg no NG: TNT 12, AN 34.5, K nitrate 33, Amm chloride 20 and moisture 0.75 parts. Maximum chge 32 oz and swing 2.62 inches (vs 3.27 for Gelignite contg 60% NG)
Ref: E. deBarry Barnett, "Explosives", VanNostrand, NY (1919), 132

Expelling or Ejecting Charge. See Vol 2 of Encycl, p C150-L, under CHARGE

Expendable. It refers to a supply item that is consumed, or loses its identity when used; expected to be consumed or to lose its identity
Refs: 1) Glossary of Ord (1959), 109-R
2) OrdTechTerm (June 1962), 144-R

Experimental Procedures in Detonation (and Explosion). See under DETONATION (AND EXPLOSION), EXPERIMENTAL PROCEDURES in Vol 4 of Encycl, p D299-R to D346-L

Experimental Procedures for Determination of Properties of Detonation Waves are discussed in Refs: 3, 32, 35, 44, 57, 67, 68, 69, 75, 94, 95a & 96 listed in Vol 4 of Encycl, pp D724-R to D730-L

Experimental Type. In order to understand the meaning of it, we are describing here *type classification* as given in Ref, pp 306-L and 307-L & R. Under this type are understood designations made by technical committee action to record the status of items of materiel from the standpoint of development and suitability for service use. Those designations and their definitions are as follows:

A) *Development Type.* Designates an item being developed to meet a military requirement. This category is further classified in the Ordnance Corps as follows:

1) *Experimental Type.* Materiel that is being developed or modified to meet approved military characteristics or to improve design or function

2) *Service Test Type.* Materiel which has performed satisfactorily in engineering tests and is authorized to be procured in limited quantity for *user test* (See Note 1 below)

B) *Adopted Type.* Designates an item which has been found suitable for its intended military purpose and which has been type classified. This category is further classified as follows:

1) *Standard Type.* Designates the most advanced and satisfactory item or assemblage that has been adopted and is preferred for procurement

2) *Substitute Standard Type.* Designates an item or assemblage which is not so satisfactory as the standard type, but is a usable substitute therefor. It may be procured to supplement the supply of the standard type

3) *Limited Standard Type.* Designates an item or assemblage that is less satisfactory than the standard and substitute std types, but which is acceptable and may be used until the supply is exhausted

C) *Obsolete Type.* Designates an item or assemblage which has been declared unsuitable for military use

Note 1: On p 310 of the same Ref is described the so-called:

User Test, which i s an evaluation test, conducted on materiel under development following satisfactory *engineering test* (See Note 2, below). The test is performed by the using agency to determine the suitability of the developmental materiel for military use. User tests are of two types:

a) *Service Test.* A test under simulated operational conditions to determine to which degree the item meets the military requirement as expressed in military characteristics

b) *Troop Test.* A test where a troop unit, equipped with appropriate numbers of the item, operates under actual or simulated field conditions to test the suitability of the item and also the adequacy of the organization, doctrine, technique, training and logistic support required for its use

Note 2: On p 107-L of the same Ref is described the so-called:

Engineering Test. It is an evaluation test of materiel under development conducted by, or under the supervision of, the technical service concerned to determine inherent structural, electrical, or other physical or chemical qualities of the item or system tested, including those of an environmental nature, designed to provide a basis for decisions as to subsequent developmental action or the suitability of the item for *user test*

Ref: Glossary of Ord (1959), pp 107-L, 306-L to 307-R and 310-L & R

Expl(s). Abbr for Explosive(s)

expl. Abbr for explosive (adjective)

expl(s). Abbr for explode(s)

expld. Abbr for exploded

Expln. Abbr for Explosion

explode(s). To be changed in chemical or physical state usually from a solid or liquid to a gas (as by sudden vaporization or rapid chemical decomposition) so as suddenly to transfer considerable energy into the kinetic form (See also Explosion)
Ref: Glossary of Ord (1959), 109-R

Exploded View. A drawing of any article or piece of equipment in which the component parts are separated but so arranged as to show their relationship to the whole
Ref: Glossary of Ord (1959), 110-L

Exploder is the same as Blasting Machine described in Vol **2** of Encycl, p 212-L. See also Blasters'Hdb (1966), pp 113–119
Note: Accdg to NOLTR **1111** (1952), p G2, exploder is an alternate term for a fuze, usually used in connection with torpedoes

Exploder. British name for an expl component contg CE (Tetryl) for use in bombs
Refs: 1) Anon, TM **9–1985–1** (1953), p 6 & others 2) H. Bullock of PicArsn Museum; private communication (1973)

Exploder, Magnetic. A magnetic exploder used by the Germans during WWI in sea mines. It contd a compass and, when a magnetic mass (such as a ship) approached, the needle of compass started to swing and this activated the initiating device of the mine

The Amer magnetic exploder of Mackie was used in torpedoes. It utilized the variations in the intensity and direction of the earth's magnetic field adjacent to the hull of a vessel to activate it in the following manner:

When the torpedo was ejected, it rushed thru the water and this spun a small turbine. This, in turn, operated a gear train that pushed the detonator into the booster cavity. The 450 yards of travel of the torpedo required for this operation gave the vacuum tubes within the mechanism time to warm up and to ready the influence feature of the device. When the torpedo entered the magnetic field created by the enemy hull, the EMF generated in the induction coils began to charge. This charge amplified by the vacuum tubes was harnessed to release the firing pin

When impact rather than magnetism was to induce explosion, the impact dislodged an inertia element which released the firing pin

The exploder mechanism weighed 91 lbs
Ref: B. Rowland & W. Boyd, "US Navy Bureau of Ordnance in WWII", USGovtPrtg-Off, Washington, DC (1953), 99–109

EXPLODING BRIDGE WIRE (EBW) or **EW** (Exploding Wire), also known as **EEW** (Electrically Exploding Wire) *(Explodierend Brückendrabt* in Ger). EBW phenomenon may be defined as the rapid disintegration (and evaporation) of fine wires under formation of a shock wave, thru abrupt application of high electric energy produced, for example, by a capacitor (condenser) discharge
Historical. Disintegration of fine wires by electrical current released by a Leyden jar (which is actually a condenser) was first observed in 1774 by Brit scientist Nairne (Ref 1), but there was no suggestion for a practical application of this phenomenon (See also Ref 7). It was forgotten until 1920, when Anderson (Ref 2) repeated the experiment of Nairne and reported that the temperatures produced on disintegration of wires approached those of the sun. He did not suggest, however, any practical application of this phenomenon, but raised an interest in it resulting in further experiments which were conducted in Europe, USA and Japan. It seems that the first application of exploding wires to initiate an explosive was done in 1938 by A.F. Belyaev of Russia, as was already mentioned in Vol **4** of Encycl, p D807-L

In Section 3, Part E, pp D807 to D810-L of Vol **4** are described EBW detonators. There are listed Ref 45 (on p D1032-R) & Ref 46a (p D1033-R) and Additional Refs: Ad97a (p D1044-L), Ad97b (p D1044-L), Ad112a (p

D1045-R), Ad141 (p D1047-R) and Ad160 (p D1052, which contains 20 refs)

The following Refs on EBW were not included in the description of EBW detonators given in Vol 4 of Encycl:

Refs: 1) E. Nairne, PhilTransRoySoc(London) **64**, 79–89(1774) 2) J.A. Anderson, ProcNatlAcadSciUS **6**, 42–3(1920) and Ibid **8**, 231–32(1922) 3) Z.V. Harvalik, "Explosive Initiators, Electric", NAVORD Rept No **1487**(1950) (A Literature Survey) 4) J.A. Anderson, "General Characteristics of the Electrically Exploded Wire", AstrophysicalJournal **64**, 295–314(1954) 5) R. DePersio, "Exploding Wire and Spark Gap Center Initiator for High Explosives", BRL MemoRept No **851**(1954) 6) W.H. Richardson, "Exploding Wire Phenomena", SandiaCorpContr **AT–(29–1)–789**(1958) 7) W.G. Grace, "Bibliography of the Electrically Exploded Wire Phenomenon", AFCRC (Air Force Cambridge Research Center) Rept **TN–58–457**(1958) 8) W.G. Chase & H.K. Moore, "Exploding Wires", Plenum Press, NY (1959) (Many refs) 9) M.T. Hedges, "Development of Exploding Bridgewire Initiators", FREL-IEAS Rept **84**(1960) 10) M.T. Hedges, "Exploding Wire Detonators", PATM **ORDBB–DR4–36**(1960) 11) M.T. Hedges, "Technical Symposium on Propellant Actuated Devices", Frankford Arsenal, 14–16 June 1961 (Discussion on EW initiators developed at PicArsn and on their requirements and advantages) 12) M.T. Hedges & E.L. Miller, "Development of an Exploding Bridgewire Ignition Element", PicArsn Ammunition Group Rept **102**(1961) (This item having the configuration of the Mk2Mod2 was developed, along with a suitable EBW firing system. Such an item was not readily susceptible to radiated RF energy) 13) F.D. Bennett, "Shock Producing Mechanisms for Exploding Wires", BRL Rept No **1161**(1962) 14) Anon, "Exploding Wires Energize Laser", C&EN, March 26, 1962, pp 43–4 [The energy obtd from an EW has been used to energize, or "pump" a LASER (See Vol 4 of Encycl, p D436-L, under "Detonation, MASER and LASER in") at Westinghouse Research Laboratories, Pittsburgh, Pa. Scientists at the American Optical Co, Southbridge, Mass have designed a system that may allow using the sun for pumping a LASER. In the Westinghouse experiment, Nichrome, Al, Cu & W(tungsten) wires were used and were exploded by electrical discharges from a bank of capacitors that is capable of an energy output of 33000 joules and up to 20000 volts. Results obtd from the work were considered encouraging] 15) H.S. Leopold, "Initiation of Explosives by Exploding Wires", NOLTR **63–159**, 15 May 1963 16) Ibid, Pt 2, NOLTR **63–244**, 15 May 1963 17) H.S. Leopold, "Initiation of Explosives by Exploding Wires III", NOLTR **64–2**, 17 Mar 1964 18) H.S. Leopold, "Initiation of Explosives by Exploding Wires IV", NOLTR **64–61**, 4 May 1964 (Effect of Wire Length on the Initiation of PETN by Exploding Wires) 19) J. Blackburn & R.J. Reithel, "Exploding Wire", 3rdProcConferExplodingWirePhenomenon, Boston, Mass (1964), **153–73** (Sweeping image photographs of the EBW initiation of PETN) 20) J.G. Pelphrey, "The Evaluation of RDX for Exploding Bridge Wire", PATR **3366**(1966)

Exploding Bridge Wire Detonators. See Vol 4 of Encycl, pp D807 to D809

Exploding Bridge Wire Detonators of Reynolds Industries, Inc. Recent addns to EBW detonators include one with pigtail leads and one coaxial. Info available on these devices includes nature of the expl materials, bridgewire, connectors, firing specifications in terms of minimum energy and potential, maximum cable length, and functioning time. Time fluctuations are within 0.02 microseconds provided that the same circuit is used

Ref: Reynolds Industries, Inc, 2105 Colorado Ave, Santa Monica, Calif 90404, quoted from Expls&Pyrots, Vol **2**, No 7(1969)

Exploding Bridge Wire Devices; Their Protection from Damage by Spurious RF Energy. Under the title "Skin Effect RF Bridge Filter", there is described an invention which provides

a much simplified approach to a device which excludes current from a load. This device can be used to protect electroexplosive systems from damage by spurious RF energy. The filter, which can be produced at very low cost, is a Wheatstone bridge or lattice network which uses resistive elements composed of a metal conductor coated with a second metal. At a predetermined frequency the filter, by utilizing the skin effect, becomes a balanced bridge which excludes current from a load

By suitable construction the invention can exclude either high or low frequency current from a load

Refs: 1) D.A. Schlachter, USP 3704434(1972) 2) Expls&Pyrots, Vol **6**, No 4 (April 1973) (Abstract of patent)

Exploding Bridge Wire Devices, Nondestructive Testing of. Several nondestructive test techniques have been developed for electroexplosive devices. The bridgewire responds, when pulsed with a safe level current, by generating a characteristic heating curve. The response is indicative of the electrothermal behavior of the bridgewire-explosive interface. Bridgewires which deviate from the characteristic heating curve have been dissected and examined to determine the cause of the abnormality. Deliberate faults have been fabricated into squibs. The relationship of the specific abnormality and the fault associated with it have been demonstrated (Ref 1, abstracted in Ref 3)

The paper of Ref 2, abstracted in Ref 3, reports a technique that will yield insight into the quality of EBW devices in a nondestructive manner. By employing a self-balancing bridge it is possible to ascertain the electrothermal and nonlinear behavior of the device. A sinusoidal current is passed thru the device which provides a signal in the form of a unique Lissajous Display (See Note, below). This display can be qualitatively evaluated and abnormal units can be readily detected

Note: Lissajous Display are figures formed by the locus of points, the horizontal and vertical coordinates of which are given by sinusoids having different frequencies, amplitudes or phases

Refs: 1) V.J. Menichelli & L.A. Rosenthal, "Fault Determinations in Electroexplosive Devices by Nondestructive Techniques", Jet Propulsion Lab Rept **TR 32-1553**, March 15, 1972. Order as N 72-20920 from NTIS, USDeptCommerce, Springfield, Virginia 22151 2) L.A. Rosenthal & V.J. Menichelli, "Electrothermal Follow Display Apparatus for Electroexplosive Device Testing", JetPropln Rept **TR 32-1554**, June 1, 1972. Order as N72-21281 from NTIS 3) G. Cohn, Expls&Pyrots, Vol **6**, No 3(1973) (Abstract of Refs 1 & 2)

Exploding Bridge Wires Energize Laser. See Ref 14 under EXPLODING BRIDGE WIRE in this Vol

Exploding Bridge Wire Initiation of Explosives. The following NOLTR's (Naval Ordnance Laboratory Technical Reports), White Oak, Maryland 20910 on "Initiation of Explosives by Exploding Wires", are listed in Expls&Pyrots, Vol **1**, No 7(1973). They are all authored by H.S. Leopold:

1) "Effect of Circuit Inductance on the Initiation of PETN by Exploding Wires", NOLTR **63-159**, May 1963 (AD 424-513) 2) "Effect of Circuit Resistance on .....," NOLTR **63-244**, May 1963(AD 431-785) 3) "Effect of Wire Diameter on .....," NOLTR **64-2**, March 1964 (AD 600-058) 4) "Effect of Wire Length on .....," NOLTR **64-61**, May 1964 (AD 601-990) 5) "Effect of Wire Material on ..... ," NOLTR **64-146**, October 1964 (AD 609-449) 6) "Further Effects of Wire Material on ..... ," NOLTR **65-1**, March 1965 (AD 463-360) 7) "Effect of Energy Termination on ..... ," NOLTR **65-56**, June 1965 (AD 618-675) 8) "Survey to Determine Explosives Capable of Initiation at Moderate Voltage Levels", NOLTR **65-127**, Nov 1965 (AD 476-199) 9) "Exploratory Studies of Explosive Initiation at High Voltage Levels" (U), NOLTR **66-144**(Conf),

September 1966 (AD 376–792) 10) "Capacitance-Voltage Relationships for the Initiation of PETN by Exploding Wires", NOLTR **66–188**, April 1967 (AD 814–149) 11) "Effect of Reduced Inductance on the Initiation of Insensitive Secondary Explosives" (U), NOLTR **67–165** (Conf), November 1967 (AD-388–810) 12) H.S. Leopold, "Effect of Crystal Habit, Density and Particle Size on the Initiating Energy of Tetryl", NOLTR **68–24**, March 1968 (AD 831–373) (The EBW initiation sensitivity to impact of Tetryl can be drastically changed by altering its crystal habit. It appears to peak at some particle size other than the smallest, and drops sharply when loading density is increased beyond point where the material no longer shows signs of settling)
Note: All of these Refs are available from Defense Documentation Center, Cameron Sta, Alexandria, Virginia 22314 or from Commander (Code 242), US Naval Ordnance Laboratory, White Oak, Md 20910

Exploding Conductors. Techniques have been developed to observe & measure the rapidly varying currents and extremely high voltages of discharge systems. It was discovered that X-rays are produced under certain conditions by exploding conductors. In addn, a time history of the X-ray emission was obtd which indicated that X-rays were emitted at times of the order of 28×10^{-9} sec after initiation of the discharge, and that radiation persisted for 8×10^{-9} sec

Spectrographic studies were also made to det the temp & densities. The plasma temp and particle densities were also detd by using the relative intensities of two adjacent lines of the same species and applying the Saha equation

In addn to temp & density measurements, spectrographic investigations were made near the UV emission produced by exploding Al wires in vacuum using the W (water-dielectric) capacitor
Ref: W.R. Faust et al, "Exploding Conductors", NRL (Naval Research Laboratory), Final Report **6009**, Oct 31, 1963

Exploding Wire (EW). Same as Exploding Bridge Wire (EBW) or Electrically Exploding Wire (EEW)

Exploding Wire Materials. A wire exploder capable of vaporizing small wires in times less than 10^{-7} sec was used to study the electrical expln of various materials & sizes in a variety of environments. Simultaneous voltage & current oscillograms with nanosecond resolution were obtd. These measurements were supplemented with time-resolved Kerr cell photographs of the wire at different times during an expln as well as by time-integrated spectrograms

The description of the electrical props of exploding wires in air prior to dwell for peak specific power levels betwn 5×10^{12} and 2×10^{14} watts/mole is as follows:
Wires of various elements can be classified in two phenomenological classes

1) the low boiling point, low heat of vaporization materials, and

2) the transition elements

In silver & copper the approach to dwell commences with the onset of vaporization. The apparent resistivity during this phase is a function only of time, and the initial wire radius is independent of the rate of energy input & wire environment

In all the materials studied, early photographs show a uniform structure for the exploding wire. A uniform illumination persists for most materials until the beginning of post-dwell conduction, although in Al shortly after expln bright spots and non-uniform regions of illumination occur

Copper (a Class 1 material) showed no observable expansion of vapors during the phase when main sequence was followed. Tungsten, on the other hand, showed appreciable expansion of vapors after the onset of vaporization and before the end of vaporization

For wires mounted in environments other than air, with other conditions remaining unchanged, the apparent resistivity as a function of specific energy density has the same dependence in the initial phase as in air

From the standpoint of technology, exploding wires are being used as fast-intense light sources, as fast fuses, as generators of fast shock waves, as sources of fast dense plasma for plasma propulsion & hypervelocity particle accelerators, as well as in various electronic switching & pulse shaping applications. Furthermore, the possibility of putting enough energy into a wire to initiate a thermonuclear reaction has been under investigation from time to time

Ref: F.H. Webb, "Study of Electrically Exploded Wire Materials", EOS Rept **210-Final**, 16 Feb 1961, Electro-Optical Systems, Inc, Pasadena, Calif (Contract DA-04-495-ORD-1298)

Exploding (or Detonating) Ammunition and Explosives Destined for Destruction. Explosive materials or ammunition items which cannot be destroyed by burning or by chemical method must be destroyed by explosion (or detonation) or drowned in deep places of the ocean as far as possible from the nearest land (See Vol **3**, p D28-L)

Destruction by burning of Black Powder is described in Vol **2** of Encycl, pp B177-R & B178-L and of Bombs on pp B233-R & B234-L. Burning Ground or Destruction Site is on pp B357-R & B358-L and Burning of Ammunition and Explosives for Destruction is on pp B358-R & B359-L. Burning of Pyrotechnic Items in Vol **3**, p D29-R. Chemical Destruction of Explosives in Vol **2**, p C173-L

Decontamination of water Containing Dissolved Explosive Wastes is in Vol **3**, p D36-L

Destruction by detonation (or explosion) of bombs in described in Vol **2**, p B234-L; of explosives in Vol **3**, p D28-R; of artillery shells (except shrapnel shells), p D28-R; of mortar shells, p D28-R; of shrapnel shells, p D28-R & D29-L; photoflash bombs, antitank mines, rockets & grenades, p D29-L; loaded metal components (such as fuzes, primers, detonators & boosters), pp D29-L & D29-R; chemical munitions, Vol **2**, p C176-R; and of pyrotechnic items, p D29-R

Alphabetical List of Destruction and Disposal Procedures for Explosives and Propellants is given in Vol **3**, pp D29-R to D35-R

In exploding (or detonating) of ammunition or explosives, the usual procedure is to place a block of TNT, Dynamite, Nitrostarch or Comp C3 in intimate contact with the material to destroy and to detonate the block with a Blasting Cap, ignited either electrically or by means of a Bickford Fuse (also known as Miner's Safety Fuse). In the first case, the electric wires should be of sufficient length to allow the personnel to retire behind a barricade out of danger; in the case of Bickford Fuse its length should be such that the time of burning is sufficient to allow the personnel to retire behind a barricade or other suitable protection before the explosion takes place

In exploding of ammunition or explosives, the following safety regulations should be observed:

1) If the destruction of materials takes place on an artillery range or similar site which is far away from magazines or other buildings, the material may be piled directly on the ground, covered with a 2-ft layer of earth, and exploded

2) If the site of destruction is located closer than 800 yards to buildings, a pit, trench or bombproof shelter should be used and the material should be covered with a thick layer of logs, earth, etc, to limit the range of fragments

3) The number of units that may be safely destroyed at one time must be carefully determined by starting with a limited number until the maximum that can be destroyed without damage to surrounding property, is determined

4) After each blast, or in case of a misfire, the personnel should not approach the site until a period of at least 30 minutes has elapsed. A careful search should be made for any unexploded material, and if any be found, it should be mixed with the material to be exploded at a later time

5) Materials awaiting destruction should be stored at a distance of not less than 500 ft, preferably in small piles located about 100 ft apart. This is to prevent accidental ignition or explosion from fragments, burning embers or grass fires

6) It is most important that personnel conducting these operations be properly trained. To send untrained personnel on such jobs would be criminal
7) Ample fire-fighting equipment should be available in the immediate vicinity and the ground should be wet down at the close of each day's operation
Refs: 1) US Army, Chief of Ordnance, "Safety and Storage Manual for Explosives and Ammunition", O.O. Form No 5994, Washington, DC (1928), Section XI, pp 4–5 2) War Department Technical Manual, **TM 9–1904**, Washington, DC (1944), pp 752–72 3) Dept of the Army Technical Manual, **TM 9–1300–203** (April 1967), Chapter 6, pp 6-1 to 6-2 (*Destruction of ammo to prevent* enemy use) 4) US Army Materiel Command Regulation **AMCR 385–100**, Safety Manual (April 1970), Chapter 27, pp 27-1 to 27-28 (Collection & Destruction – explosives, ammunition, chemical agents & munitions)

Exploration for Natural Gas and Oil by Seismic Method (Geophysical Prospecting). It is known that the earth has been segregated by geological processes into layers of various density and elastic behavior. It is also known that natural gas and oil are formed in sedimentary areas and in the normal course of events they gradually migrate towards the surface of the earth until they are trapped by some dense, relatively impermeable structure or by irregularities in layering, such as faults and folds which may serve as reservoirs for accumulation of gas or oil. In the early days of oil prospecting, the surface indications, geological studies and "hunches" provided the only bases on which the oil-men could select a drill-site. Since about 1923, however, increasing use has been made of subsurface maps obtained by *geophysical methods*. The most important of these methods is *seismic exploration (or prospecting)*. It is based on the fact that a sudden shock, such as that caused by an explosion sends out seismic energy in all directions. This energy is divided when it reaches the boundaries of geological formations of different acoustic impedances (the density multiplied by the sound velocity), while some of the energy returns to the surface where it is detected by extremely sensitive electromechanical instruments, known as geophones. When these instruments are placed near the source of expln, so as to record the energy travelling in an essentially vertical path, the technique is known as the *reflection method*. When the geophones are extended over large distances from the source, as compared to the depth of interest, the technique is known as the *refraction method*. In this case the seismic energy is propagated over an essentially horizontal path in a high-velocity material overlain by a low velocity material. The vibrations returning to the surface are picked up by geophones. They convert the ground motion into electricity which is amplified and recorded by an oscillograph and magnetic tape recorder. The oscillogram, commonly known as the *record*, permits an immediate inspection to determine if the shot were properly recorded. The magnetic tape record is subsequently analyzed at a data processing center where geological maps are prepared (Ref 2, pp 351–52)

More detailed description of the refraction method is given on pp 353–54 and of reflection method on p 354

Accdg to Meyer (Ref 1, p 401), expls used in seismic exploration must be especially water-resistant, very powerful to give expl waves which will be readily detectable over large areas within the limits of the instruments used, and have a high detonation velocity. Blasting Gelatin and Gelatin Dynamites have been used for their power. For detonators it is required that they not only fire the charges but the exact instant of detonation of the charge must also be known. In using an Electric Blasting Cap, there is some short lag between closing the electric circuit and detonation of the charge; in other methods the delay is even greater. This problem, known as that of the time lag, can be solved by studying characteristics of carefully standardized Electric Caps and by some other methods. At the time of publication of Ref 1 (1943), Hercules Electric Caps were

said to have a time lag of 0.0047 seconds when fired singly at 1.5 amperes with a maximum deviation of 17% and an average deviation of 6%. Accdg to the curve showing average firing time versus current for the DuPont "SSS" Seismograph Electric Blasting Caps (See Ref 2, p 363), the time lag at 1.5 amperes current is about 2.5 milliseconds (0.0025 seconds). Explosives recommended by the DuPont & Co include: Seismograph 60% "High-Velocity" Gelatin, "Nitramon" WW, "Nitramon" WW-El, "Nitramon" S, "Nitramon" S-El, "Nitramite" WW, "Nitramite" WW-EL, "Seismogel" A, "Seismex" and "Seismex" PW (Ref 2, pp 358–62)

Refs: 1) M. Meyer, "Explosives", Th.Y. Crowell Co, NY (1943), 401 2) Blasters'-Hdbk, 15th Ed (1966), Chap 23, pp 351–366

Explorer Series of Artificial Earth Satellites. This series consisting of several satellites was sponsored in 1956 by the US Army Ballistics Agency and later in conjunction with the National Aeronautics and Space Administration (NASA). Other scientific, engineering and academic institutions assisted. Work began on the program in April 1956, and by the end of January 1958, Explorer 1 was lifted into orbit. It was the USA's first and the world's third artificial satellite to be successfully orbited. Of the seven satellites sponsored before 1960, five were successfully launched: Explorer 1, 3, 4, 6 and 7

The first five satellites were very similar; cylindrical in shape, length approx 6ft, 6 inches, diam 6 inches, pay load 30.8 lbs, instruments 11 lbs, perigee attained approx 225 miles, apogee 1575 miles. The carrier vehicle, the Juno 1. Explorer 6 was spheroidal in shape with 4 solar cells mounted on protruding paddles or vanes which extended 3 ft out from the main body. Length of the satellite was 2 ft,42 inches, diam 26 inches, pay load 142 lbs, perigee 156 miles, apogee 26360 miles. Due to the size and weight, the carrier vehicle Thor-Able 3, Explorer 7 had a shape of two joined truncated cones. Its length was 2.5 ft, diam 30 inches, pay load 70 lbs,perigee attained 346.6 miles, apogee 15945 miles. The carrier vehicle was Juno 2.

The purpose of the series was to accumulate scientific data, such as inside, outside and skin temperatures; density, distribution and momentum of micro-meteorites, cosmic-ray intensity energy and intensity of corpuscular radiations, X-radiation, the extent of the earth's radiation belt, map the earth's magnetic field, etc

Note 1: Perigee is the point in the orbit of a satellite of the earth that is nearest to the earth

Note 2: Apogee is the point in the orbit of a satellite of the earth at the greatest distance from the center of the earth

Ref: F.I. Ordway & R.C. Wakeford, "International Missile and Spacecraft Guide", McGraw-Hill, NY (1960) (United States of America/70–75; New Developments/12)

Exploseur électrique. French for Electric Exploder

Explosibility and Flammability of Explosives, Propellants and Pyrotechnic Compositions. See "Detonability and Flammability of Explosives, Propellants and Pyrotechnic Compositions" in Vol 4 of Encycl, pp D211-R to D213-R and also "Detonability of Propellants" in Vol 3, p D103-L

Addnl Refs: A) E. Oehley, ChemIngeTech **25**, 399–403 (1953) & CA **47**, 10851 (1953) (Explosibility equations) B) Akira Suzuki, JIndExplosivesSoc, Japan **14**, 142–63 (1953) & CA **49**, 11281 (1955) (Explosibility of initiators by heat and impact) C) J.F. Shaw, USBurMines, Bull **551**, 11 (1955) & CA **49**, 15241–42 (1955) (Explosibility characteristics of mine-fire atmospheres; a graphic method of determining) D) W.A. Grey & A.L. Bennett, JChemPhys **23**, 1979–80 (1955) & CA **50**, 2174 (1956) (Explosibility by shock waves) E) P. Tavernier, MémPoudres **36**, 231–35 (1954) & CA **50**, 2980 (1956) (Explosibility by shock waves) F) V.J. Clancey, "Results of Explosibility Tests on Various

Organic and Inorganic Compounds", Royal Armament Research and Development Establishment, GtBritain, RARDE Report (X) 6/63

Explosif(s) Belgian and French Explosive(s), whereas the word "explosive" in an adjective

Explosifs à l'aluminium. Fr for Aluminized Explosives. Several Fr formulations are listed in Vol 1 of Encycl under ALUMINUM CONTAINING EXPLOSIVES, on p A146-L. Examination in 1902 by CSE (Commission des Substances Explosives) showed that some of these expls, as, for example, Formula 226 (p A146-L), were more powerful than PA (Picric Acid). More recently (1948 & 1950), Médard (p A148-L) determined the properties of the following Fr aluminized explosives: Nn°30, Nn°31, Nn°32, Nn°33, 63–CSE–1949, Sofranex A and Sevranite n°1

Explosifs d'amorçage. Fr for Priming or Initiating Explosives. No information at our disposal about compns of such Fr expls

Explosif amylacé. See Amide (Explosif) in Vol 1 of Encycl, p A168-L

Explosif antigrisou. See Antigrisou (Explosif) in Vol 1, p A466-R

Explosifs antigrisouteux. French industrial explosives authorized for use in fiery coal mines, because when used under prescribed conditions they do not ignite mixtures of methane-air-coal dust. They may be subdivided into *explosifs couche* (qv) and *explosifs roche* (qv)

Corresponding explosives are known in Belgium as *explosifs SGP* (sécurité-grisou-poussière), and in the USA as *permissible explosives*. In Great Britain they are called *permitted explosives*, and are to be distinguished from *authorized explosives* which conform to certain conditions with respect to safety in handling, in transport, etc. Both kinds, permissible and authorized, are *safety explosives*, known in France as *explosifs de sûreté* and in Germany as *Sicherheitssprengstoffe*. The Ger name for permissible expls is *Schlagwettersichere Sprengstoffe* and are distinguished from *Handhabungssichere Sprengstoffe*, the term applied to those which are safe to handle. In Russia, permissible expls are known as *Antigrisutnyiye* or *Predokhranitel'nyiye Vzryvchatyiye Veschestva*, in Spanish speaking countries as *explosivos de seguridad contra el grisú* and in Italy as *esplosivi antigrisutosi*

Refs: 1) Marshall 2(1917), 582 2) Davis (1943), 347 3) Pérez Ara (1945), 250 4) Belgrano (1952), 165 5) Yaremenko & Svetlov (1957), 113

Explosifs brisants (or détonants). Fr for High Explosives

Explosifs C or Explosifs du type C. See C (Explosifs) in Vol 2, p C1-L

Explosifs chloratés, also known as Explosifs O or Explosifs du type OC. See Chlorate Cheddites in Vol 2, pp C155 to C159

Explosifs couche. Fr expls with temp of expln below 1500°, which are permitted for use in coal layers of fiery coal mines. For example, Grisou-naphthalite couche contains AN 95 & TNNaphthalene 5%, while Grisou-dynamite couche contains NG 12.0, collodion cotton 0.5 & AN 87.5% (Ref 1). Sartorius and Kreyenbuhl (Ref 2) reported *Explosifs couche* contg NaCl 15 to 25% and *Explosifs couche améliorés* contg 44 to 58% NaCl (Comp with Explosifs roche)

Refs: 1) Davis (1943), 350–51 2) R. Sartorius & A. Kreyenbuhl, MP 38, 90 (1956)

Explosifs CSE. See CSE (Explosifs) in Vol 3 of Encycl, pp C570-R & C571-L and also Explosif 123–CSE–1948, Explosif 136–CSE–1948 and Explosif 68–CSE–1949 in Vol 3, p C451-L

Explosif "D". Fr high explosive: RDX 85 & TNT 15%
Ref: C. Fauquignon et al, 4th ONR Symposium of Detonation (1965), p 45-R

Explosifs DD. See DD Explosifs in Vol 3 of Encycl, p D19-L

Explosifs déflagrants. Fr for Low Explosives (LE)

Explosifs de démolition. Fr for Demolition Explosives

Explosifs détonants (or brisants). Fr for High Explosives (HE)

Explosif E. Swiss explosive, received at Picatinny Arsenal from GtBritain and examined by Livingston. Its compn was found to be AN 77, Nitroguanidine 19 & hydrated magnesium nitrate. It could be cast at 111–13°, but was of low brisance
Ref: S. Livingston, PATR **907** (1938)

Explosifs Favier. See FAVIER EXPLOSIVES in Vol 6 of Encycl

Explosif "Formule 63–CSE–1949". Fr expl: AN 67, Pentolite (PETN 80 & TNT 20%) 12 & Al 21%. Power (CUP) 147
Ref: L. Médard, MP **32**, 215 (1950)

Explosif Formule 226. See Vol 1 of Encycl, p A146-L

Explosifs fulminants. Fulminating Explosives such as MF

Explosif Le Maréchal (Poudre Le Maréchal): K chlorate 84 & stearic acid 16%
Ref: Gody (1907), 266

Explosifs de mine. Fr for Mining (or Commercial) Explosives

Recent progress and actual tendencies in the field of mining explosives in France was discussed by L. Médard in MP **32**, 209–24 (1950)

Fr "nonpermissible" mining expls are listed in Vol 3 of Encycl, p C438-L & R

Fr "permissible" coal-mining expls are listed on pp C450-R & C451-L

Explosif MDN (or MDn). A Fr HE consisting of Dinitronaphthalene 1 & PA 4 parts. It had a lower mp (105–10°) than PA (120–22°) and was less sensitive to shock. It was used for cast-loading projectiles
Ref: Davis (1943), p 157

Explosif MDPC. Fr HE contg PA 55, Dinitrophenol 35 & Trinitro-m-cresol 10%. Its low mp (80–90°) permitted its use for cast-loading projectiles
Ref: Davis (1943), p 166–67

Explosif MMN. A Fr HE contg PA 70 & Mononitronaphthalene 30%, fused together under water. It was used as a bursting chge in drop bombs during WWI
Ref: Davis (1943), p 157

Explosif MMTC. A Fr yel HE consisting of PA 55, TNT 35 & Dinitrophenol 10%. Because of its low mp (80–90°), it can be easily cast loaded. It was used as a bursting chge for shells
Ref: Davis (1943), p 166

Explosif Müller. Older Belg grisoutite, manufd at Baelen-sur-Nèthe: NG 46, Mg sulfate 45 & woodflour 9%
Ref: Gody (1907), 357 & 705

Explosifs N or du type N, also known as **Explosifs nitratés** (Nitrate Explosives). Fr mining expls contg AN. They can be nonpermissible and permissible. Accdg to Marshall, Vol 1 (1917), 389, they are also known as *Grisounites*. They can be either couche (See Explosifs couche) or roche (See Explosifs roche)

Examples of older Explosifs N are listed

in Marshall 1(1917), 388, Barnett(1919), 141 and Davis(1943), 367; whereas newer formulations are given in Refs
Refs: 1) R. Sartorius, MP **32**, 145(1950) 2) A. LeRoux, **33**, 265–82(1951) 3) L. Médard & A. LeRoux **34**, 195–204(1952) 4) This Encycl, Vol **3**(1966), p C438-L & R lists the following nonpermissible Explosifs du type N: Nn°0, Nn°1b, Nn°1c, Nn°1d, Nn°20, Nn°21, Nn°30, Nn°31, Nn°32, Nn°33. In the same Vol, p C450-R & C451-L are listed: Explosif du type Nn°7 (of Burlot & Schwob), Nn°9 (of Audibert) and Nn°62 (of Médard)

Explosifs N (nitratés) à l'aluminium (Nitrate Explosives with Aluminum). See under ALUMINUM CONTAINING EXPLOSIVES in Vol 1, pp A146-L & A148-L

Explosif NDNT: AN 85, TNT 5 & DNNaphthalene 10%. This explosive, as well as Explosifs: **NT** (AN 70 & TNT 30%), **NTN** (AN 80 & TNNaphthalene 20%), **N2TN** (AN 50, Na nitrate 30 & TNNaphthalene 20%) and **NX** (AN 77 & TNXylene 23%) were used in France during WWI for loading 75mm shells in lieu of Schneiderite, known as Explosif Sc
Ref: Davis (1943), 367

Explosifs à la nitrocellulose. In addition to using straight slightly wet Guncotton in compressed form for loading some shells and torpedo warheads (as was done in France to a limited extent during WWI and in Russia as late as WWI), the French also used the so-called *potentite*, which consisted of CP_1 (NC of 13% N) & K nitrate 50%. They also used the *mélange de Sarau et Vieille*, which contd CP_1 40 & AN 60%. Another mixture proposed by the same scientists contd CP_1 58 & K chlorate 42% but was too dangerous to use. The Belgians used a mixture consisting of CP_1 52 & Ba nitrate 48%
Ref: Pepin Lehalleur (1935), 354

Explosifs N à la pentolite (Nitrate Explosives with Pentolite). The following expls were developed in 1942 by Lécorché and their manuf began in 1945:

Nn°20: AN 78.2, Pentolite (20/80) 18.3 & woodflour 3.5%; its power by CUP 119.5 (PA 100)

Nn°21: AN 75.4 & Pentolite (80/20) 24.6% (CUP 123.5)
Ref: L. Médard, MP **32**, 214–15(1950)

Explosifs NR, Explosifs du type NR or Explosifs nitratés résistant à leau (Nitrate Explosives Resistant to Water). In order to render some AN mining expls resistant to water and to prevent caking of AN particles, Médard (Ref 1) and LeRoux (Ref 2), suggested coating of expl mixtures with small amts (0.5–1.0%) of Ca stearate. The use of this substance in expls was first proposed in 1896 by G. Roth of Germany and later patented by duPont & Co (USP 2048050 of 1934). The Commission des Substances Explosives (CSE) studied not only the use of stearate (as waterproofing agent), but also the abietate, naphthenate, resinate, alignite, etc but came to the conclusion that Ca stearate gave the best results. It was found, however, inefficient for aluminized expls (Ref 1)

The following samples prepd at LaPouderie d'Esquerdes were examined by LeRoux (Ref 2):
Nn°0R: AN 79, TNT 20 & Ca stearate 1%. Power by CUP 117 (TNT 100)
Nn°1cR: AN 87.5, DNNaphthalene 11.5 & Ca stearate 1%. CUP 109
Nn°7R: AN 76, DNN 7, woodmeal 1, NaCl 15 & Ca stearate 1% (CUP 84)
Nn°21R: AN 75.5, Pentolite (20/80) 23.5 & Ca stearate 1%. CUP 121
Nn°30R: AN 81, TNT 9.5, Al 8.5 & Ca stearate 1%. CUP 135
Nn°31R: AN 79, Pentolite (20/80) 11.5, Al 8.5 & Ca stearate 1%. CUP 134
Refs: L. Médard, MP **32**, 216–17(1950). 2) A. LeRoux, MP **33**, 265–82(1951)

Explosif NT
Explosif NTN
Explosif N2TN
Explosif NX
See above under Explosif NDNT

Explosifs O or OC (Explosifs du type O). Cheddite-type expls authorized in France before WWII for use in coal mines. They existed in several formulations as can be seen from Tables 2, 3 & 5 of Vol **2** of Encycl, pp C157 & C159

Explosif O2: K chlorate 79, DNT 15, MNNaphthalene 1 & castor oil 5%
Ref: Barnett(1919), 111

Explosifs O3 or Prométhées were authorized at the end of the 19th century in France under the name of **explosifs On°3** for blasting purposes. They also were authorized in Italy. They were developed in 1897 in Russia by Ievler and were considered as *Sprengel-type Explosives*. Explosifs On°3 were prepd by dipping cartridges of a compressed oxidizing mixture of K chlorate 80–95 & Mn dioxide 20–5% into a liquid prepd by mixing nitrobenzene, turpentine and naphtha in the proportions 50/20/30 or 60/15/25. The most serious disadvantage of these expls was an irregularity in behavior resulting from the circumstance that different cartridges absorbed different quantities of the combustible oil, generally betw 8 & 13% and that the absorption was uneven and sometimes caused incomplete detonation
Refs: 1) Daniel (1902), 382 2) Marshall **1** (1917), 379 3) Davis(1943), 355 4) Clift & Fedoroff **2**(1943), p P14 5) Pérez Ara (1945), 231 (Explosivo Prometeo) 6) Giua, Trattato **VI**(1)(1959), 397(Prometeo)

Explosif O4 or Explosif P: K chlorate 90 & paraffin 10%; deton vel 3565m/sec at d 1.45
Ref: Barnett(1919), 111

Explosif O5: Na chlorate 79, DNT 16 & castor oil 5%
Ref: Barnett(1919), 111

Explosif On°6B Minélite B: Expl mixture consisting of K chlorate 90, paraffin 7 & vaseline 3% used by the French during WWI in grenades and mines. A similar expl contg Na chlorate 90, instead of K chlorate, was used in grenades and trench mortar bombs
Note: Minélite A contd K chlorate 90, paraffin 7 & heavy petroleum oil 3%, while *Minélite C* contd K chlorate 89, paraffin 5, vaseline 4 & pitch 2%
Refs: 1) Davis(1943), 361 2) Pérez Ara (1945), 210

Explosif P (potasse). Same as Explosif O4, listed above
Note: Accdg to Vandoni, MP **22**, 149(1926), the same term was applied to mixture of Amm perchlorate 61.5, Na nitrate 30.0 & paraffin 8.5%

Explosifs perchloratés. See Perchlorate Cheddites in Table 5, C159 of Vol **2**. It includes among other mixtures the French Cheddites I, II & III and Sevranites n°1 and n°2. One Belgian Cheddite (known as Yonckite), one British (known as Blastine), one Swedish (known as Territ), one Ger military Cheddite and three German Perchloratites are also included

Explosifs plastiques. The following French plastic expls are listed in Vol **3**, p C438; Explosif du type n°18, Nobélite, Sevranite No 1, Sevranite No 2, Sofranex A and Tolamite. See also L. Médard, MP **32**, 217–19(1950)

Explosifs et Poudres. Fr expls and propellants patented after WWII by the État Français, FrP 971644(1951) & CA **46**, 9311(1952) include mixtures of combustibles, such as: NGu (Nitroguanidine), HNDPhA (Hexanitro-

diphenylamine) and 9-Oxo-2,4,5,7-tetranitrothiodiphenylamine with oxidizers, such as nitrates, chlorates or perchlorates. For example, mixtures of NGu 53 with Amm perchlorate 47% and mixts of NGu 56 with K nitrate 44% were claimed to possess high temperatures of deflagration and be suitable for use in weapons operating at high temperatures or for oil seismic prospecting at great depth

Explosifs roche. Fr expls with temp of expln betwn 1500° and 1900°, which are permitted for use in rock layers of fiery coal mines. For example, Grisoudynamite roche contains NG 29.0, collodion cotton 1.0 & AN 70.0%, while Grisoudynamite roche salpetrée contains NG 29.0, collodion cotton 1.0, K nitrate 5.0 & AN 65.0%
Ref: Davis (1943), 351

Explosif S (soude): Na chlorate 89 & paraffin 11%
Refs: 1) Davis (1943), 361 2) Pérez Ara (1945), 210

Explosif Sc. Fr designation of **Schneiderite**, which was prepd accdg to Davis (Ref 1) by incorporating 7 parts of AN and 1 part of DNNaphthalene in a wheel mill, and was loaded by compression. It was used by the French during WWI for loading small- and medium-sized HE shells, especially 75mm

Pérez Ara (Ref 2) gives compn of Schneiderite as AN 88, DNNaphthalene 11 & resin 1%
Refs: 1) Davis (1943), 367 2) Pérez Ara (1945), 240

Explosifs SGP (Explosifs sécurité-grisou-poussière). Accdg to Marshall (Ref 2), "explosifs SGP" stand for Belgian term "explosifs sûr-grisou-poussière" and signify explosives safe (sûr) to be used in coal mines contg firedamp (grisou) and dust (poussière). Testing of commercial expls intended for use Belgian fiery coal mines was formerly conducted at Frameries gallery, near Mons, by the method briefly described in Ref 2, p 585, and more fully in Ref 1, pp 710–11. The current gallery is located at Pâturages and the tests are briefly described in Ref 3, pp C710-R & C711-L. If no ignition of firedamp-dust mixture takes place in the gallery with maximum charge ("charge limite") of 31 oz, the expl is qualified as SGP, but max chge allowed underground is only 28 oz
Refs: 1) Gody (1907), 710–11 2) Marshall **2**(1917), 585 3) Vol **3** of Encycl (1966), pp C370-R & C371-L

Explosifs au silicium. Under this term are known expl formulations contg pulverized silicon, developed in 1950 at the laboratories of the Commission des Substances Explosives. They were "explosif n°78–CSE–1950" and "explosif n°88–CSE–1950". Their compositions and properties are given in Vol **3** of Encycl, p 571-L

Explosifs de Sprengel. Dr H. Sprengel, the inventor of the mercury pump, took out in 1871 patents for a series of mining expls to be made by mixing oxidizing substances with combustibles in such proportions that their oxygen balance to CO_2 should be close to zero. The essential feature was that the two constituents were to be mixed together on the spot just before the blasting operation and the mixture was to be exploded by a fulminate detonator. As oxidizing agents Sprengel recommended fuming nitric acid and K chlorate; as combustibles many substances, among them nitrobenzene, nitronaphthalene, petroleum, carbon bisulfide and PA. Expls using nitric acid did not find much use because they were corrosive and dangerous to handle; and if the acid came in contact with a detonator, a premature explosion could take place (Ref 3, p 43)

Gody (Ref 2) listed several mixtures using nitric acid, among them HNO_3 71.9 & NBenz 28.1%; HNO_3 60 & DNBenz 40%; and mixture known as **Oxonite**: HNO_3 41.7 & PA 58.3%

More successful than Sprengel nitric acid expls, were his mixtures of K chlorate with

liquid combustible materials. Gody (Ref 2) listed one of such mixtures as K chlorate 90, carbon bisulfide 9 & PA 1% and stated that it was very brisant

The American S.R. Divine patented in 1880's several such mixtures, among them *Rack-a-rock* used on Oct 10, 1885 for the great blasting operation of Hell Gate in New York Harbor (Ref 3, p 43). These expls are described in Vol **5** of Encycl, p D1525-L & R

Many Sprengel expls based on chlorates are described under CHEDDITES OR STREETITES in Vol **2** of Encycl, pp C156-R to C159, Tables 1, 2, 3 & 4. Most of the formulations listed in these tables are French

One of the Sprengel-type expls was developed in 1897 in Russia by Ievler and authorized in France for blasting purposes under the name of *Explosif On°3* (qv) or *Prométhée*

Accdg to Marshall (Ref 3, p 43) there are considerable advantages to transporting separately two inert ingredients, as oxidizers and combustibles of Sprengel expls, but against this must be put the difficulty and inconvenience of mixing the constituents in the right proportions on the spot. If made up beforehand, the cartridges are dangerously sensitive and become more so on keeping. In case of K chlorate expls, the chlorate is made up into porous cartridges which are transported to the spot of blasting, where they are dipped into liquid combustibles just before use. These expls were not allowed in Great Britain, as this dipping operation is considered to constitute a process of manufacture, and consequently could only be carried out in a duly licensed factory (Ref 3, p 378)

More detailed description of Sprengel expls is given by Davis (Ref 5, pp 353–60), who lists among Sprengel or Sprengel type explosives: *Oxonite* (See above), **Hellhoffite** (1879): HNO_3 72 & NBenz 28%; *Rack-a-rock* (See above); *Explosif On° 3* (or Prométhée) (See above); **Fielder's** – a liquid contg NBenz 80 & turpentine 20 parts absorbed by a mixture of K chlorate 70 & K permanganate 30 parts; **Kirsanov's** – a mixture of turpentine 90 & phenol 10 parts absorbed by a mixture of K chlorate 80 & Mn dioxide 20%; *Panclastites* of Turpin are described below under Explosifs de Turpin; *Anilithe* (See Vol **1**, pp A442-R & A443-L); *Oxyliquits* prepd by impregnating porous cartridges of combustibles with liquid oxygen or liquid air, just before blasting operation; and chlorate and perchlorate explosives (See also Ref 4, pp 11–12)

Refs: 1) Daniel (1902), 736–37 2) Gody (1907), 709–11 3) Marshall **1** (1917), pp 43 & 378–83 4) Stettbacher (1933), pp 11–12 & 309 5) Davis (1943), 353-60

Explosifs de sûreté (Safety Explosives). French and Belgian term for expls "safe to handle and to transport", but it does not mean that such expls are safe for use in coal mines with atmosphere contg firedamp and coal dust. For expls safe to use in such coal mines, the term "explosifs antigrisouteux" is applied. This distinction between the terms was recommended by Belg Prof Watteyne and adopted by the Congrès de Chimie Appliquée at Rome in 1906 (Refs 1a & 2)

Older "explosifs de sûreté" are described by Daniel (Ref 1)

A current definition of "explosifs de sûreté" is given in the French "Réglement pour le Transport des Matières Dangereuses" as follows: An "explosif de sûreté" is an explosive which does not present any danger of explosion in mass either by combustion or by shocks (impacts) of the highest intensity which may be produced in the course of transportation and handling (See Ref 6, p 213, footnote)

The term "explosifs de sûreté" corresponds to Amer and British "Safety Explosives"; German "Handhabungssichere Sprengstoffe" (Refs 3 & 4); Russian "Besopasnyiye v Obrashchenii Vzryvchatyiye Veshchestva" (Ref 5); Italian "Esplosivi di sicurezza" (Ref 2) and Spanish "Explosivos de seguridad" (Ref 4)

Refs: 1) Daniel (1902), 747–60 1a) Gody (1907), 719 2) Molina (1930), 294–95 3) Davis (1943), 347 4) Pérez Ara (1945), 250 5) Yaremenko & Svetlov (1957), 12 6) L. Médard, MP **32**, 213 (footnote) (1950)

Note: It is stated on pp 213–14 & 223 of Médard that after WWII, the *explosif de sûreté*

Nn° 1d had been used in French mining. It contained AN 89, DNNaphthalene 9 & wood-flour 9% and replaced "explosifs Nn°1b and Nn°1c" listed on p C438-L

Explosifs et Poudres de Turpin. Accdg to Daniel (Ref 1, p 777), the following expls and propellants were invented by Eugène Turpin beginning in 1881: *Boritine* (See Vol **2** of Encycl, p B250-R); *Celluloidine* (See Vol **2**, p C95-R); *Duplèxite* (See Vol **5**, p D1570-L); **Fluorines** (Permissible expls patented in 1888: 1) NG 37.5, kieselguhr or rhodanite 12.5 & Ca fluoride 50.0%; 2) K chlorate or nitrate 35, Ca fluoride 50 & DNBz 15%) (Daniel, p 302); **Mélinite** [Expl mixture of PA 70 & NC (previously dissolved in acetone) 30%, patented in 1890's for use in torpedoes and demolition charges (See Daniel, pp 434–36) (The term mélinite is also applied to straight PA)]; **Oxydine** [Safety expl patented by Turpin in 1888. It was a mixture of Dynamite No 1 50 with ZnO or ZnS 50%. Dynamite No 1 consisted of NG 75 & kieselguhr 25% (Daniel, 594)]; **Panclastites** [Liquid, Sprengel-type expls patented by Turpin in 1881. They consisted of nitrogen tetroxide (which is a less corrosive oxidizer than strong nitric acid) and a liquid combustible substance, such as carbon bisulfide in proportion 1:1.81 which corresponds to equation:

$$CS_2 + 3NO_2 \rightarrow CO_2 + 2SO_2 + 3N.$$

Other liquid combustibles, such as petroleum, benzene, toluene, etc, were proposed, but Turpin preferred carbon bisulfide. Panclastites were successfully used in Belgium as military demolition explosives and in Germany they were tried for loading projectiles. For this two glass containers with thin walls were used. One was filled with NO_2 and the other with CO_2. They were placed inside the projectile, one on top of the other, and in order to prevent premature breakage, several rubber cubes were inserted between the walls of projectiles and glass containers. The glasses broke by shock on departure of projectile from thegun barrel and this caused the mixing of two ingredients. Explosion of mixture took place on arrival to target (See Daniel, pp 596–98)]; **Panclastites-guhr** [Solid expls patented by Turpin in 1882. They consisted of Panclastites absorbed by solid, porous materials, such as kieselguhr. They were less powerful than corresponding liquid expls (See Daniel, p 598)]; **Picramic Acid** and its salts were patented by Turpin in 1887 & 1888 for use in expl mixtures, listed in Daniel, p 615; **Picric Acid (PA)** and **Picrates** were patented in 1885 by Turpin for use in some expl mixtures. This was not new, because PA was already used as early as 1867 by **Borlinetto** (See Vol **2** of Encycl, p B250-R); **Progressite** (Powder patentented by Turpin in 1888: Ba nitrate 65, Amm picrate 15, DNBz 10, coal tar 6 & brown charcoal 4%. It could be ignited without the use of a detonator) (See Daniel, p 651)

Note: Other Progressites listed in Daniel, p 652 were safety expls which were not invented by Turpin. They will be described under P's

Pyrodialytes. Under this term Turpin patented, beginning in 1881, chlorate expls using tar (goudron) as a combustible. In order to neutralize acids present in tar, 2 to 10% of Na carbonate or bicarbonate was incorporated. They are described by Daniel on pp 661 to 663

Following Table Ex1 lists nonpermissible Pyrodialytes (Compositions are given in parts because they do not always add up to 100)

Table Ex1

Components	Pyrodialytes			
	Extra Forte n°0	Forte n°1	Lente n°2	Lente n°3
$KClO_3$	88	80	40	40
$NaNO_3$	–	–	48	–
KNO_3	–	–	–	40
Tar(Goudron)	10	10	20	20
Charcoal	5	6	5	–
K (or NH_4) bi-carbonate	2–3	4	4–5	4–5

Table Ex2 lists some perchlorate Pyrodialytes patented in 1898, (in %)

Table Ex2

Components, %							
$KClO_4$	80	60	–	–	–	–	–
NH_4ClO_4	–	–	85	75	60	50	40
$KClO_3$	–	–	–	–	15	–	40
Tar	10	10	15	10	10	10	10
NH_4TNCresylate	10	–	–	–	–	–	–
NH_4TNPhenate	–	30	–	–	10	–	10
AmmTNBenzoate	–	–	–	15	–	–	–
GuN*	–	–	–	–	–	40	–
NH_4MnO_4	–	–	–	–	5	–	–

* Gu = Guanidine

Other "explosifs de Turpin", listed in Daniel include:

Turpin (Poudres dites â double effet). These expls patented in 1881 & 1883 contained K chlorate about 80 & coaltar about 20 with some charcoal and may be considered as precursors of "Pyrodialytes". It was allowed to replace half of chlorate by K or Pb nitrate or to replace 10% of chlorate by K permanganate. Some of the formulations contd silicium or kieselguhr if tar were too fluid (pp 777–78)

Turpin's Explosives patented in 1887 contd as main ingredients chloro-, bromo-, or iodonitrates of coal tar without inorganic oxidizers. They could be initiated by a MF detonator or by a cartridge of Dynamite (p 778)

Turpin's Explosive, patented in 1883 contg Ba nitrate 60, DNBz 15 & MNPhenol 25% could be initiated by a safety fuse (p 778)

Turpin Explosives with Percarbonates, especially with $K_2C_2O_6$, were suggested because they evolve on decompn of oxygen and can explode on being initiated by a very strong detonator, even if they are not mixed with other substances. Their use in admixture with Pyrodialytes was recommended (p 778)

Turpin's Propellant, described by Gody (AddnlRef A, p 641) patented in 1888, was prepd by incorporating into NC jelly some camphor and paraffin which served for slowing down the rate of burning. After passing thru a macaroni press and cutting into desirable sizes, the grains were dried and packed into cartridges

Turpin's Explosive, described by Colver (AddnlRef B) was patented in 1885 for filling HE shells. It was prepd by mixing powdered PA with small amounts of collodion cotton, grease, or aqueous gum arabic and forming into blocks of the desired shape and size

Note: Accdg to Stettbacher (AddnlRef C, 15), Turpin discoverdd in 1885 that PA (Picric Acid) can easily be detonated by means of a MF cap and for this reason can serve as pressed or cast chge for filling HE's shells. It was used for this purpose in France since 1886 under the name of *Mélinite* and in Gt-Britain since 1888 under the name of *Lyddit* and then later in Japan as *Shimose*. Although PA was known for more than 100 years before Turpin's discovery, it was not used straight but always in mixtures, such as of *Borlinetto* and of *Désignolle* invented in 1867 (See Vol **2** of Encycl, p B250-R and Vol **3**, p D90)

Addnl Refs for Explosifs de Turpin:

A) Gody (1907), 266, 270, 551–52 & 641 B) Colver (1918), 14 C) Stettbacher (1933), 12, 15 & 182 D) Pepin Lehalleur (1935), 245, 255, 344 & 359 E) Davis (1943), 166 F) Pérez Ara (1945), 211, 226, 228 & 513 G) Giua, **VI** (1) (1959), 403 (Panclastiti)

EXPLOSION. See under DETONATION, EXPLOSION AND EXPLOSIVES, Introduction and Definitions in Vol **4** of Encycl, pp D217 to D223-L (11 refs)

Various items described in Vol **4**, pp D225 to D732 under Detonation should also be applied to Explosion

Explosion of Ammoniacal Silver Compounds and Their Solutions are described in Vol **1** of Encycl, pp A305 & A306-L with 8 refs. An abstract of a recent "Safety Dept Report on Ammoniacal Silver Nitrate Explosion" by

R.R. Whitman, Oct 1968 is published in Manufacturing Chemist Assoc, March 1969, Accident Case History No 1554. Also, an abstract by J.A. Conklin is found in Expls-&Pyrots 3(4)(1970). Whitman's rept discusses in depth the potential hazard of ammoniacal silver salt solutions. The solns of Ag salts in Amm hydroxide are occasionally encountered in synthetic inorganic chemistry and, in addition, are frequently employed as "Tollen's reagent" for detn of the presence of aldehydes *Note:* Tollen's reagent is prepd by dissolving 10g of silver nitrate in 60ml concd ammonia & 40ml water; a small quantity of this soln is mixed immediately before adding to the sample in a test tube with an equal vol of 10% aq NaOH soln. The formation after a while of a silver mirror in a test tube indicates the presence of aldehydes

Whitman's rept described several cases where laboratory prepn of ammoniacal Ag salt solns led to explns. In addn, quotations from the literature sources demonstrate the variety of warnings (or lack of the same) that have been given for Ag salt-ammonia systems. The majority of workers discussed in this rept appear to favor the theory that Ag_3N (silver nitride or "fulminating silver") is the explosive species involved. Precipitation of a dark material prior to expln was reptd in several of the cases cited by the author

Preventive measures and a list of literature references are included in the report

For addnl info on Fulminating Silver, see Vol 6 of this Encycl

Explosion Autocatalytic. See Vol 4 of Encycl, pp D225-R to D226-R

Explosion, Break Character of. See Vol 4, p D226-R

Explosion, Breaking Theory of Carl. See Vol 4, p D227 to D228

Explosion, Bulge Test, used to evaluate welding methods is briefly described by C.G.Dunkle, Lecture on Detonation, delivered at Picatinny Arsenal, Session 26, 23 April 1958, p 234. More detailed description is given by P.P.Puzak, in "Explosion-Bulge-Test. Performance of Low-Carbon Ni-Cr-Mo-B Quenched and Tempered Steel Weldments", NRL Rept 4919, May 17, 1957

All of the weldments prepd for this investigation were carefully ground smooth in accordance with procedures reqd for 100% X-ray inspection of pressure vessels

Test conditions previously established for other 1-inch quenched & tempered steel weldments of the same strength level as Y-steel (a proprietary low alloy steel contg less than 1% each of Ni, Cr, Mo etc) were followed in the expln bulge test of Y-steel weldments. Thus, 20 x 20 inch weldments were explosion loaded using 7-lb Pentolite chges placed at 15-inch offset distance from the test sample. Generally, expln bulge testing is discontinued after 3 successive shots if failure is not developed or after that shot which results in a Visible indication of failure. For this test program, it was recognized that microfissures were present in the weld metal as received. Accordingly, several of the Y-steel weldments were tested with an addnl explosive shot even though crack indications were obtd on the second shot

Expln bulge tests were conducted at 30°, 0°, and -50° F. For "general service" application (pressure vessels, tanker or cargo ships) it was deemed that Y-steel weldments would perform satisfactorily at all ambient temps. It was clear also that this material is not suitable for applications based on "military service" loading conditions (submarine hulls reqd to withstand expl action of depth charges)

Explosion, Chain Reactions. See Vol 4, p D229

Explosion of Coal Dust and/or Firedamp. See under Coal Mine Explosions and Fires in Vol 3, pp C360-R to C367-R

Explosion of Coal Gas. See Firedamp Explosion under Coal Mine Explosions and Fires in Vol 3, pp C360-R to C367-R

Explosion in Coal Mines. See previous item

Explosion of Combustible Substance and Transition from Combustion to Detonation. See A.F. Belyaev, ZhPriklKhim **23**, 432-39 (1950) & CA **47**, 2987 (1953) and also in Vol 4 of Encycl under Detonation (and Explosion) Development (Transition from Burning (Combustion) or Deflagration, pp D245-R to D252-R (35 refs)

Explosion and Combustion Research at the Hungarian Mining Research Institute, Budapest. The Paper of Jósef Száva is published in German language under the title "Explosions- und Verbrennungs forschungen in Ungarischen Forschungsinstitut für Bergbau" in Polish journal "Archiwum Procesów Spalania" (Archives of Combustion Processes), Vol 1, No 1, pp 49-53 (1970). On pp 52-3 are listed 11 references and on p 53 are given Polish and Russian abstracts

Following is an abbreviated translation by Mr Cyrus Gl Dunkle, formerly of Picatinny Arsenal and now residing at 2500 Holman Ave, Silver Spring, Maryland 20910:

It is known that initiation of solid TNT to explosion cannot be caused by local heating to high temperature, unless the material contains some bubbles of gas, such as air, Ar, etc. This means that the presence of gas bubbles is of importance, although they do not seem to take part in the chem reaction. The luminescence of these gases on shock heating plays the decisive role. By 2-color pyrometry with electronic amplification the temp exceeded 5000°K during the 1st few μs of the 20-30-μ sec process, far above what could be due to the reaction heat. Adiabatic compression by the shock waves heats the entrapped gases intensely, and their radiation is largely absorbed in the explosive. For further clarification, the change in the entire radiation spectrum with time was observed, and microdensitometric measurements were made at points corresponding to 1,5,75, and 200μs after initiation in cast and in powd TNT. At 1st the detonation spectrum is that of extremely hot gas (500-6000°K) under high pressure. Later, up to 75μ sec, the spectrum is that of a high-pressure gas and a solid body together. The luminosity increases up to 75μsec and then decreases. At this point most of the reaction is completed and the material, up to this time at high pressure and high d, has absorbed most of its own radiation

Thermodynamic treatment of the processes occurring through the shock waves traversing the explosive showed that the greater the ratio of the energy evolved on the break-up of the explosive mol (E_q) to the activation energy (E_a), the less energetic the shock needed to initiate an explosion. If the ratio is low, as in many explosives, good initiation can be insured by suitable porosity and introduction of gas bubbles.

Radiation energy also palys a role, heretofore overlooked, in the initiation of firedamp by mine explosives. The IR absorption spectra of explosion-suppressing additives and the intensity/time relationship of the inflamation were recorded. The best suppressors were those of moderate B,P, or F content. They were effective even if 8-10% less were used than of the more usual materials, and did not impair either the stability or the performance of the explosive

The ignitibility of fire-damp by ignition cartridges was reduced by the presence of CO_2 and mositure in the mine gases but increased by the presence of combustible gases and by heating. Raising of pressure at 500-800 torr had no effect. An industrial Dynamite-type explosive giving better fire-damp-safety was developed contg limestone powd instead of the usual rock salt or AN. The storage, stability, or explosive performance were not affected. The limestone powd with sp surface of 3000cm^2/g absorbed heat not only by its own temp rise, but also by thermal dissocn. Its lack of hygroscopicity is another advantage. Whereas the brisance of Dynamite contg limestone and AN decreases about 20% on damp storage for 500 hr, that of Dynamite contg limestone alone,

actually showed a small improvement, 10%, which was not diminished on normal storage for 1-2 yr. When like amts of limestone powd, rock salt, and AN were added to Dynamite batches, that contg limestone had the greatest brisance. Andreev attributed this effect to suppression by the limestone of the formation of active intermediate explosion products which initiate explosion of CH_4/air mixts. Also, their formation decreases the brisance

In researches for prediction, prevention, and control of spontaneous mine fires, O absorption on exposed coal surfaces in certain proportions, along with moistening, had an adverse effect and thru a fire barrier not thick enough set into motion air streams capable of building up ignition centers. In quasi-adiabatic oxidn, researches with measurements of rate consts and apparent activation energies in oxidn processes at low temps with isothermal O absorption, the changes in the O-contg functional groups of the bituminous-coal mol (OH, COOH, CO and CH_3O), as also the effects of their concns on O-absorbability and the tendency toward self-ignition, were studied. With a coding system developed by the "contact period" or "induction period" detn method, bituminous and brown coals can be characterized from the viewpoint of their tendencies toward oxidn and toward self-ignition

Explosion, Combustion and Shock Waves. See Combustion, Explosion and Shock Waves in Vol 4, p D172

Explosion of Composite Explosives. See Vol 4, pp D232-R

Explosion of Composite Propellants. See Vol 4, p D238

Explosion of Condensed Explosives. See Vol 4, pp D238-R to D242-L

Explosion, Craters in. See Vol 4, p D242-R

Explosion, Damage Caused by. See Vol 4, p D245-L

Explosion, Dangers (Hazards) of. See Vol 4, pp D245-L and D366 & D367

Explosion, Dangers in Chemical Plants by. See Vol 4, p D245-L

Explosion, Dark Waves in. See Vol 4, p D245-L

Explosion, Decay in. See Vol 4, p D245-L

Explosion, Development from Combustion or Deflagration. See DETONATION (AND EXPLOSION) DEVELOPMENT (TRANSITION) FROM BURNING (COMBUSTION) OR DEFLAGRATION in Vol 4, p D245-R to D252-R (35 refs)

Explosion and Fire Suppression Techniques. Pressure-time relation for aircraft fuel mixts show that max press requires a stoichiometric mixt as well as max pressure before explosion. Rate of pressure rise is dependent on flame speed, vessel size, and nature of the ignition source. Three types of detectors are used to control such expls. Two are visual detectors, one detects rate of pressure use. After detection, expln is suppressed within msecs by expln of a capsule contg 2–8ml per gal of $CBrF_3$, CBr_2F_2, CH_2BrCl, CCl_4, or CH_2Br
Ref: G. Grabowski, SAE Trans **63**, 803–8 (1955) & CA **49**, 16435 (1955)

Explosion Disasters, Prevention of. The most useful terms for evaluating the hazards of various combustible-oxidant

systems are the limits of flammability and min spontaneous ignition temp. A review of terms used in gas explosions technology is followed by a discussion of many ways of preventing disasters
Ref: M.G. Zabetakis & G.W. Jones, ChemEngProgr **51**, 411–14(1955) & CA **49**, 15242 (1955) (Prevention of industrial gas explosion disasters)

Explosion, Distant Effect of. See Vol **4**, p D252-R

Explosions of Dusts. See Vol **5**, pp D1578-R to D1579-R

Explosion of Dusts, Mists and Vapors. See Vol **4**, pp D253-L to D255-R

Explosion, Dynamics of. See Vol **4**, pp D255-R to D256-L

Explosion, Effects of Blast and Shock Waves on Structures. See Vol **4**, pp D256 & D257

Explosion, Effects of Inert Components on. Detailed measurements and calculations are made of the energy transferred to chemically inert "fillers" by physical processes such as heat conduction and compression during the detonation of TNT. Further evidence was obtained that the temp coeff of the processes controlling reaction rates in the detn is small
Ref: J.L. Copp & A.R. Ubbelohde, TrFaradSoc **44**, 646–69(1948)

Explosion, Effects of Inert Gases on the Explosion Delimitation of Methane-Air Mixtures. The origin, development and end of explns of methane-air mixts (I) are discussed according to the relation of the explosion nos. Formulas are given for the calcn of the expln delimitation of I under the influence of inert gases. Mathematical results are compared to Jones' empirical results. First practical application of the new math method is also described
Ref: F. Otásek, Uhlí **5**, 382–90(1955) & CA **50**, 11017–18(1956)

Explosion, Effects of the Physical Structure and the State of Aggregation on the Detonating Capacity of Explosives. Liquid TNT detonates only if the diam of the specimen is above 32mm. TNT powder compressed to the sp gr of the liquid TNT, detonates at diams of 2.1mm or greater. Pieces of solid TNT are similar to liquid TNT. Maximum diams for liquid TNT and TNT powder are 2.3 and less than 2mm, respectively. Gas space between expl particles facilitates deton, presumably because hot gases spread the deton
Ref: A.Ya. Apin & V.K. Bobolev, ZhFizKhim **20**, 1367–70(1946); Engl abstract in CA **41**, 3297(1947)

Explosion, Effects Produced in Air, Earth and Water. See Vol **4**, pp D257-R & D258-L

Explosion, Effects of Sheaths on Gases from. Sixteen permissible expls, 7 of which were in cartridges of 2 sizes, 1¼x8 inches and 1½x8 inches. Nine different sheaths were tested at low loading densities in one application and with confinement and fireday stemming in another. Results indicate that sheathed expls do not introduce important hazards from prod of CO and in some cases amt is reduced by their use. Undesirable amts of N oxides may be produced by too great a wt ratio of sheath to expl
Ref: E.J. Murphy, USBurMines Repts Invest **4220**, 57pp(1948) & CA **42**, 4348(1948)

Explosion, Electrical, Electromagnetic and Magnetic Effects Accompanying It. See Vol **4**, pp D258-L to D264-L

Explosions of Electrostatic Origin in Industry, Prevention of. Explosions during electro surgery of the colon can be caused by intestinal CH_4 and(or) H_2. Expts with H_2 show introduction of CO_2 thru a smoke-aspirating sigmoidoscope at the rate of 2 l/m will inhibit the explosions
Ref: E.I. Levy, AmJSurg **88**, 7548(1954) & CA **49**, 1186(1955)

Explosions, Elements of Risk in. The elements of risk in incendiaries and expls and their prevention are discussed
Ref: A. Blandin, Rhône: A. Martez & Givors, 368pp (1952) & CA **47**, 6657 (1953)

Explosion, Energy of. See Vol **4**, pp D264-R to D266-R

Explosion, Enthalpy in. See Vol **4**, p D265-R under Explosion, Energy of

Explosion, Enthalpy Excess in. See Vol **4**, pp D267-L to D268-L

Explosion, Entropy in. See Vol **4**, p D265-R under Explosion, Energy of

EXPLOSION, EQUATIONS OF STATE IN. See Vol **4**, pp D268-L to D298-R (Numerous refs)

Explosion, Expanding-Jet Theory of Jones. See under Detonation (and Explosion), Nozzle Theory of Jones, Vol **4**, pp D460-R to D461-R

Explosion, Experimental Data Interpretation of. See Vol **4**, p D299-L

EXPLOSION, EXPERIMENTAL PROCEDURES. See Vol **4**, pp D299-R to D346-L (Numerous refs)

Explosion Hazards from Use of Coal Dust. See Coal Dust Explosion Hazards from Its Uses on p C359-R of Vol **3**

Explosions and Fires. See Fires and Explosions

Explosion, Gap Tests in. See List in Vol **4**, pp D303-R & D304-L

Explosions – Gaseous, Kinetic Studies of. Flash photolysis techniques are described, the reaction products being observed in both emission and absorption with resolution times of about a μsec. Some results with C_2H_2–O mixts photo sensitized with NO_2 are given
Ref: R.G.W. Norrish et al, 5th Symp on Combustion Pitts **1954**, 651-6 (1955) & CA **49**, 16437 (1955)

Explosions in Gases. See Vol **4**, pp D351-L to D360-L

Explosions in Gases. Determination of Ignition Points. See Vol **4**, p D360

Explosions of Gases, Vapors and Dusts. Development (Transition) from Burning (Combustion) or Deflagration. See Vol **4**, pp D360-R to D363-R

Explosion in Granular Explosives. See Detonation in Granular Explosives in Vol **4**, pp D365-R & D366-L

Explosion Hazards (Dangers) of. See Vol **4**, pp D366 & D367 and also p D245-L

Explosion Hazard of Organic Vapors, Reduction of. A process which comprises the treatment of wood with hydrocarbon vapors at an elevated temp, after which compressed air is used to impregnate the wood with a preservative. Expln hazards are reduced by introducing steam at 10–20psi before applying the air. Steam is added in an amt equal to the partial pressure of the organic vapor in the closed space

Ref: M. Hudson, USP 2633429 (1953) & CA **48**, 5502 (1954)

Explosion by Heat, Flame and Spark, Initiation. See under Detonation (and Explosion) Initiation and Propagation in Explosive Substances in Vol 4, pp D402-L to D417-R

EXPLOSION; HEATS OF FORMATION, COMBUSTION, DEFLAGRATION, EXPLOSION AND DETONATION. See Vol 4, pp D369-L to D384-R

Explosion, Heat Sensitization of Explosives and Memory Effect. See Detonation, Heat Sensitization of Explosives and Memory Effect on pp D367-R to D369-L of Vol 4

Explosions, High Altitude Effects. High altitude nuclear explns have helped considerably in understanding natural radiation belts. The Argus artificial radiation belt made in 1958 gave info on the stability of particle orbits. The Starfish artificial belt made in 1962 gave quantitative info on electron lifetimes not available any other place. Below L = 1.7 the electrons have a long life and apparently are lost only by coulomb scattering on the atmosphere. Above L = 1.7 the lifetime shortens abruptly and some non-atmospheric process dominates. Synchrotron radiation & aurora were also observed after the Starfish nuclear expln

Ref: W.N. Hess, "The Effects of High Alti - tude Explosions", National Aeronautics and Space Administration, NASA **TND-2402**, Sept (1964)

Explosions, History of is reviewed in the book of R. Assheton, published by the Institute of Makers of Explosives, NY (1930)

See a detailed discussion of this subject under History of Explosives in Vol 7 of Encycl

Explosions, Homogeneity by Flash Photolysis. Light emitted from flash-initiated explns of acetylene and O photo sensitized by NO_2 in a large quartz tube was studied photoelectrically. Presence of very short-lived intense emission from the diat radicals was confirmed. Although main part of expln is homogeneous, 2 deton waves travel a short distance to the ends of the reaction vessel. Sharp peak is caused by the impact of one of these deton waves on the vessel window nearer the spectrograph

Ref: B.A. Thrush, ProcRoySoc (London) **A233**, 147–51 (1955) & CA **50**, 4595 (1956)

Explosions by Hot Wires. Ignition failures with thin nichrome wires in methane-air were due to fusion of the wire before sufficient energy had been communicated to the gas to effect ignition

Refs: 1) H.P. Stout & E. Jones, 3rd Symp on Combustion and Flame and Explr Phen, University of Wisconsin, Madison, 329–36 (1948) & CA **46**, 11624 (1952)

Explosions in Ice. Twenty-four instrumented and 106 uninstrumented blasts ranging in wt from 2.5 to 40 lbs of four types of explosives (60% Straight Gelatin, Composition C4, Atlas Coalite 7S & Atlas Coalite5S) were detonated above, in contact with, and at various depth below ice surfaces

These tests were conducted at the edge of the Greenland Ice Cap, near Camp Tuto, during the summer of 1957. This study was designed to obtn fundamental data on producing excavations in frozen ground, on forming trenches & foxholes for troops in arctic & sub-arctic regions, and on the design of structures in an arctic or sub-arctic environment to resist damage by explns

Under-ice direct shock pressure, air-induced under-ice shock pressure, and air-blast pressure from shallow under-ice bursts, contact bursts, and near-contact bursts were measured. The apparent crater and the true crater were measured, and the mechanics of the fracture were studied. The height to which the flyrock was thrown was measured using motion pictures, and the ground rise & venting phenomena were studied also. Sixty-six seismic records were taken

The details of this test program are given in the Ref

Ref: C.W. Livingston, "Explosions in Ice", US Army Snow Ice and Permafrost Research Establishment, Technical Report **75**, December 1960 (Corps of Engineers, Wilmette, Ill)

Explosion by Impact. See Vol **4**, p D391-R and Fig 45 on p D392

Explosion, Impetus and Available Energy of. See Vol **4**, p D391-R

Explosion, Impulse in. See Vol **4**, p D393-L & R. Included is definition of Isp-specific impulse employed in jet propulsion

Explosion, Impulse Effects on Solids. New method for recording is described by W.E. Bron et al, ASTM Bull **50** (TP 38), Feb 1957

Explosion, Impulse Specific. See Vol **4**, p D394-L

Explosion, Induction Period in Initiation of Explosives and Propellants. See Vol **4**, pp D394-R & D395-L and V.V. Voyevodskii (Voevodsky), "On Some Reactions Occurring During the Explosion Induction Period", 7thSympCombstn, 34–40 (1959) (32 refs)

EXPLOSION (OR DETONATION) BY INFLUENCE OR SYMPATHETIC DETONATION AND TESTS. See Vol **4**, pp D395-R to D402-L (Numerous refs)

EXPLOSION (AND DETONATION), INITIATION (BIRTH), AND PROPAGATION (GROWTH OR SPREAD) in EXPLOSIVE SUBSTANCES. See Vol **4**, pp D402-L to D417-R

Explosion, Initiation and Shock Processes. See Vol **4**, pp D417-R to D419-L

Explosion, Jet-Piercing Theory of. See Detonation (and Explosion), Penetration or Jet-Piercing Theory of Apin in Vol **4**, pp D467-R to D470-L

Explosion at Lake Denmark Naval Ammunition Depot Located Adjacent to Picatinny Arsenal, Dover, New Jersey.

As in all walks of life, industry has its accidents & disasters. That there are not more of them is due in a large measure to safety rules and their enforcement. The risk in the manuf & handling, and in the storing of expls has been made a special study by the Institute of Makers of Explosives and others. The American Table of Distances, a result of their studies, has been incorporated in a number of states in the USA

The activities at Picatinny Arsenal have been separated to comply with safety distances and all known means are employed to safeguard workers. When accidents have occurred, it has been due to these precautions that injuries to employees and loss of material or damage to buildings have been kept to a minimum at Picatinny

The story of the Lake Denmark Explosion that levelled many buildings at Picatinny Arsenal on 10 July 1926 is well told in the "History of Explosions", published under the direction of the Institute of Makers of Explosives. Their rept reads, in part, as follows:

On July 10, 1926, there occurred a series of explns on the Lake Denmark Ammunition Depot of the United States Navy (about 3½ miles from Dover, NJ). This Depot comprised an area of 461 acres of land, partly overgrown by trees and brush. It included approx 160 buildings, 44 of which were used for the storage of high explosives, smokeless powder, projectiles, Black Powder, the remainder being store houses, industrial buildings which did not contain expls, and dwellings

On the afternoon of July 10, 1926, there was a severe electrical storm at Lake Denmark. At about 5:15 PM lightning struck in the southwest end of the Depot, and almost immediately thereafter, black smoke was seen issuing from the northeast side of Temporary Magazine No 8. An alarm of fire was sounded and the personnel of the Depot responded immediately to the fire call, and at least one stream of water was playing on the fire when at 5:20 PM a tremendous detonation occurred at the scene of the fire

The first expln occurred in Temporary Magazine No 8, and was followed in about 5 minutes by the expln of the contents of Temporary Store House No 9, which was about 150 ft in the clear from No 8. Enormous craters were made by the two explosions. Buildings in the immediate vicinity of Temporary Store Houses Nos 8 & 9 were ignited by embers, missiles or direct heat from the explns, and the fire spread rapidly to other magazines, store houses, and shell houses

Temporary Store House No 8 contained:

2106 – 50 lb cast TNT depth charges, detonators removed
850 – aerial bombs, TNT center section
1800 – 300 lb depth charges, TNT middle
100 – 25 lb Navy Mark 1, loaded and plugged (aircraft); accessories for bombs in wooden boxes

Estimated total amt of expls 670000 lbs

Temporary Store House No9 contained:

1250000 lbs Grade A flake TNT in 50-lb boxes
350000 lbs Grade A TNT stored in 100-lb boxes
150 to 160 Mark IV aircraft bombs, probably 250 lbs TNT to each bomb

Estimated total amt of expls over 160000 lbs TNT

A third series of explns occurred in Shell Store House No 22. The contents were:

40 – 14" Class B shell, loaded & fuzed
80 – 14" AP shell, loaded & fuzed
360 – Mark 1 bombs, each loaded with 50 lbs TNT
1000 – Mark II bombs, each loaded with 50 lbs TNT
300 to 400 – Mark III bombs, loaded with TNT
200 – Mark IV bombs, loaded with TNT
500 – Mark V bombs, loaded with TNT
10000 – 8" shell, loaded & fuzed
1000 – 5" shell, loaded & fuzed

Estimated total amount of expls in building 180000 lbs

Within 3000 ft radius of Temporary Store Houses Nos 8 & 9, everything on Lake Denmark Depot was wrecked, burned or otherwise destroyed, with a few exceptions. Beyond the 3000 ft radius, and as far as the Navy reservation (estimated under 5600 ft), all of the bldgs were damaged in more or less degree by the expln, but as there were no expls or powder stored in the bldgs in this area, no fires occurred

Probably one of the outstanding matters of interest was that the following expls stored did not detonate, but burned:

2500000 lbs Explosive D stored in Temporary Store House No 11, about 500 ft away from the site of expln
300000 lbs Explosive D and 20000 lbs Picric Acid in Dry House No 1, 750 ft away from the site of expln
300000 lbs Explosive D in Dry House No 2, 300 ft from the site of expln and only 50 ft from Dry House No 1
510000 lbs TNT in bulk burned in Temporary Store House No 7

Adjoining the Lake Denmark Depot was the Picatinny Arsenal of the US Army which lay about 150 ft below the point of the hillside which was the site of Temporary Magazines Nos 8 & 9. Approximately the slope of the ground on Lake Denmark Depot continued thru the Picatinny Arsenal down to Picatinny Lake. Bordering the Lake the ground was practically level, to the west the ground rises abruptly to a higher elevation than the hills

east of Lake Denmark Depot. The level land of Picatinny Arsenal was fairly well cleared, except the east end, and the west hillside were wooded. The area occupied by Picatinny Arsenal was approx 3 miles long by ¾ mile wide, and scattered over it were the 350 or so buildings of a miscellaneous character, which comprised the Arsenal

Practically all of the bldgs on PA were placed at a lower elevation than the buildings in which the explns occurred and, as there were no barricades, were directly exposed to the force of the explns, except for such protection as was afforded by trees surrounding the location

When the explns occurred in Temporary Store Houses Nos 8 & 9, a wave of tremendous pressure was thrown over the Picatinny area intensified probably by the hill back of Buildings 8 & 9. The wave broadened out as it proceeded until it finally struck the hills to the west, and how it reacted from then on is difficult to establish, but coming or going it spread over the entire valley causing destruction or damage

Eight buildings within the 1000 to 2000 ft zone were completely demolished. Nineteen others were wrecked or considerably damaged. Thirty-five bldgs within the 2000 to 3000 f t zone were partially wrecked or seriously damaged. Buildings within the 3000 to 4000 ft zone were variously affected depending upon the materials of construction, wooden frame bldgs being completely wrecked. Substantial bldgs outside the 4000 ft zone suffered practically no damage except broken windows & cracked plaster. About 75 other bldgs, less substantial, suffered some roof damage and/or collapsed walls. Damage to outside properties in nearby communities was limited to broken windows, damaged roofs, and structural damage to frame buildings up to 3¾ miles away

Killed and Injured. Nineteen casualties were caused by the detonations. The greatest loss of life occurred in the fire-fighting parties in the vicinity of Temporary Store Houses 8 & 9. Eleven marines and one enlisted man of the Navy, and four commissioned officers were killed at this point. Most of the men killed were in the first fire-fighting party to arrive at the scene, and were probably all within 300 to 500 ft of the expln

Thirty-eight officers and privates in the Navy Marine Corps were injured. About 25 of these were with the second fire-fighting party which, with a horse cart, had reached a point near Store House No 1 (within 1000 ft of Temporary Store House No 8) when the first expln occurred. The body of a woman was found under the ruins of a two-story dwelling which stood about 300 ft from Temporary Store Houses Nos 8 & 9. One woman passenger in an automobile a short distance outside the Lake Denmark gate when the first expln occurred was so badly injured by flying debris that she died in the hospital later

The Commander of the Arsenal, Major N.F. Ramsey, and a private were at Temporary Store House No 3 (about 600 ft from Temporary Store House No 8) at the time of the first expln. Both were badly shocked & injured by flying debris, but managed to remain on duty in the upper end of the Lake Denmark Depot most of the night following the disaster. An officer, First Lieutenant George W. Bott Jr, US Army, was killed in the Beater House (Building No 385) at Picatinny by being crushed when the building collapsed. This bldg was located about 1500 ft away from the first expln

Citations for bravery were awarded the following personnel of Picatinny Arsenal for their heroism at the time of the Lake Denmark disaster. Receiving the Soldiers' Medal for heroism were:

Major Herman H. Zornig
Captain Joel G. Holmes
Captain John P. Harris
Technical Sergeant Christian J. Vogt, all of the Ordnance Dept;

US Army

Mr E. Williams
Mr I. Yamin
Mr T.E. James, all civilian employees
Major Augustus L.L. Baker, Marine Corps and contract surgeon for Picatinny Arsenal

Staff Sergeant Archie L. Downey, Finance Dept, PA and
Major Norman F. Ramsey, Ordnance Dept, Commanding Officer at Picatinny Arsenal

The New Picatinny. Immediately after the Lake Denmark catastrophe, which did terrific damage to the Arsenal 10 July 1926, no building escaping entirely and some being completely destroyed, the Chief of Ordnance, then Major General C.C. Williams, appointed a Board of Officers to appraise the damage and to make recommendations as to action that should be taken. This Board recommended not only that the Arsenal be rebuilt, but that addnl land be purchased to enlarge and improve certain portions of it. Congress in session in the fall of 1926 approved the rebuilding of the Arsenal and, in December 1927, appropriated $2300000 for this purpose

The "Old Picatinny" had been a gradual development of many years. In laying out the "New Picatinny", the reservation was divided into geographical areas, into which were placed the various activities depending upon their character. The separation of divisions was as follows:

1) manufacturing activities where powders & expls are handled

2) storage areas in which powders & expls are housed

3) manufacturing activities where non-hazardous materials are handled, and where administrative & engineering functions are carried out

The rept concluded that Picatinny is not just another Army Post, nor is it simply another explosives plant that has gotten back onto its feet after a blow. It is both of these and more because it is the very essence of our entire national defense scheme. It is the Ammunition Arsenal of the nation, the design & development of artillery ammunition for the Army being the responsibility of Picatinny

Refs: 1) Dr Kausch, SS **21**, 210(1926) (Das Explosionsunglück im Lake Denmark Naval Ammunition Depot) 2) H.S. Deck, "Safety in Explosives Plants", Army Ordn **7**, 33–37 (1927) 3) Anon, "Heroes of Picatinny Disaster Honored", ArmyOrdn **8**, 145(1927) 4) Editorial, "The New Picatinny", ArmyOrdn **8**, 380–81(1928) 5) Colonel J.K. Crain, "The New Picatinny", ArmyOrdn **9**, 328–29 (1929) 6) Captain J.A. Rogers Jr, "The History of Picatinny Arsenal", Dover, NJ (31 March 1931), Chapter IX, "Safety and the Lake Denmark Explosion", pp 72–91

Explosion, Lateral Dispersion (Expansion) and Lateral Loss in. See Vol **4**, pp D421-R to D423-L

Explosion, Lateral Rarefaction Wave. See Vol **4**, p D364-R under Detonation, Geometrical Model Theory of Cook

Explosion, Light (Flame) Accompanying It. See Detonation (and Explosion) Luminosity (Luminescence) Produced on in Vol **4**, pp D425-L to D434-L (21 refs)

Explosion Limits in Condensed Explosives. See Vol **4**, p D424-L & R

Explosion Limits Determination from a Unified Thermal and Chain Theory. In the paper of Gray & Yang (Ref 1), a mathematical model was proposed to unify the chain and thermal mechanisms of explosion. It was shown that the trajectories in the phase plane of the coupled energy and radical concentration equations of an explosive system will give the time-dependent behavior of the system when the initial temperature and radical concentration are given. In the 2nd paper of the same investigators (Ref 2), a general equation for explosion limits (P–T relation) is derived from a unified thermal and chain theory and from this equation, the criteria of explosion limits for either the pure chain or pure thermal theory can be deduced. For detailed discussion see Refs

Refs: 1) B.F. Gray & C.H. Yang, JPhysChem **69**, 2747(1965) 2) C.H. Yang & B.F. Gray, "The Determination of Explosion Limits from a Unified Thermal and Chain Theory", 11th-SympCombstn(1967), 1099–1106

Explosion of Liquid Explosives. See cross-refs in Vol 4, pp D424-R & D425-L and R.F. Chaiken, AstronauticaActa 17, 575–87 (1972) (On the Mechanism of Low-Velocity Detonation in Liquid Explosives)

Explosion, Luminosity (Luminescence) Produced on. See Vol 4, pp D425-L to D434-L

Explosion, Mechanical Effects of. See Vol 4, p D441-L

Explosion, Mechanisms of. See Vol 4, p D441-L & R

Explosion in Media of Variable Density. See Vol 4, p D441-R

Explosion, Memory Effect. See Vol 4, p D368-L & R, under Detonation, Heat Sensitization of Explosives and Memory Effect

Explosion, Mikhel'son-Rayleigh Straight Line in. See Rayleigh-Mikhel'son Line on p D354-L, Fig A and equation 9 of Vol 4

Explosion, Mining Effects. See under Blast Effects in Earth described in Vol 2, pp B182-R & B183-L and also under Crater, Cratering and Cratering Effect in Vol 3, p C553-L & R. Cratering Effect Tests are described on p D554-L & R

Explosions in Mines. See Coal Mine Explosions and Fires in Vol 3, pp C360-R to C367-R

Explosions in Mines from Al, Mg or Steel Sparks. See H. Schultze-Rhonhof, RevInd-Minérale 39, 276–90 (1957) & CA 51, 13353 (1957)

Explosions in Mines, Review. Some researches on explosions in mines after WWII and a review of progress in Belgium, France, Great Britain and the USA are given by H.F. Coward, TransInstMiningEngrs (London) 105, 228–41 (1946) & CA 40, 6259 (1946)

Explosion in Mixing House of a Fireworks Plant. An unusual explosion demolished a mixing house while two building wings remained in tact. Construction consisted of stone walls (1m thick) and a light roof. Cause was believed to be spontaneous combustion
Ref: E. Banik, Explosivstoffe 1956, 190

Explosions in Narrow Tubes, Selfacceleration of Spark-Ignited. When a CH_4–O mixt in a tube is ignited by a spark, the flame travels at the normal speed and then accelerates. In a long tube detonation may then be set up, while in a short tube the speed decreases as the end of the tube is approached. Exptl evidence suggests that initial acceleration is due to the change from a spherical to a cylindrical flame front. Expansion of hot gases then pushes the flame front down the tube, the distance traveled by the flame being proportional to the cube of the time. If gas mixt has s low burning velocity, the flame will eventually travel at a lower constant speed, which is equal to the product of the burning velocity and the expansion ratio. Rapidly burning flames, however, will give rise to a shock wave which will accelerate the flame and may lead to detonation
Ref: A. Everet & G. Minkoff, Fuel 33, 184–91 (1954) & CA 48, 6127 (1954)

Explosion, Nitrogen Reaction in. Apin et al (Ref) investigated reactions of RDX, LA & Hydrazine Azide produced on heating with powdered B, Be, Al, Mg & Zr. It was found that heat of detonation was higher than when the expls were heated alone. This was because B, Be, Al, etc interacted on heating with nitrogen of the expls with formation of

nitrides, such as BN, Be_3N_2, AlN, Mg_3N_2 & Zr_3N_4 which released additional heat
Ref: A.Ya. Apin et al, ZhFizKhim **32**, 819–22(1958) & CA **52**, 21108(1958)

Explosion, Nozzle Theory of Jones. See Vol **4**, pp D460-R to D461-R

Explosions, Nuclear (or Atomic). See Atomic (or Nuclear) Explosion in Vol **1**, p A501-R and Addnl Refs in Vol **4**, p D461-R

Explosion-Operated Tool for driving steel pins or bolts into masonry, concrete or steel was proposed by H. Behrend, USP 2835894 (1958)

Explosion Parameters and Characteristics. See Vol **4**, pp D463-L to D464-R

Explosion, Particle Size Effect in. See Detonation, Particle Size Effect in. See Vol **4**, pp D465-L to D466-L

Explosion, Penetrating Jet Theory of Apin. See Vol **4**, pp D467-R to D470-L

Explosion, Phenomena Accompanying It. See Vol **4**, p D471-L & R

Explosion Phenomenology. Accdg to Dr VanDolah (Ref) explns are generalized as the result of a rapid release of pressure without regard for its source. They may include a confined thermal expln, a confined deflagration, a detonation, or simply pressurized gas. Each of these sources is examined in broad terms and some basic similarities of the physical processes are suggested
Ref: Dr Robert W. VanDolah, "The Phenomenology of Explosions", in ProcSymp on Inherent Hazards of Manufacturing and Storage in the Process Industry, Nov 13–14, 1969, The Hague, Netherlands, Preprint, 22pp (1971) (Abstracted in Expls&Pyrots **5**(5) (1972)

Explosion Physics. The cited Ref deals mathematically with the cumulative effect of shaped charges and the jets formed from metal linings of such chges

The cumulative effect is an appreciable increase in the local action of the expls. This effect is obtd by using charges which have a cavity on one of the ends – a cumulative cavity. If such a chge is initiated from the opposite end, then the expl effect in the direction of the cavity axis is found to be much greater than if ordinary charges are used. It was established experimentally that if the surface of a cumulative cavity is covered with a relatively thin metal facing, then the armor-piercing action of the cumulative chge is increased many times
Ref: F.A. Baum, K.P. Staniukovich & B.I. Shekhter, FizikaVzryuva, Chapt **12**, 469–554, Moscow(1959) [English translation by US Joint Publication Research Service, PA Tech Note **FRL–TN–94**, Nov 1961]

Explosion, Physics of. G.I. Pokrovskii in "FizikaVzryva" (Physics of Explosion), Moscow (1955) (See Engl abstract by G.K. Kudravetz, Major, USAF, OTIA 1450, Available at PicArsn Library as No U63039) gave a discussion on an electrical explosion such as caused by a ball of lightning. Such a ball usually originates when lightning strikes some obstacle. In such case there appears a blindingly brilliant fiery ball which is easily carried by wind and which emits a unique hissing sound and is apparently saturated with certain internal movement. If this travelling ball meets any obstacle that retards this internal movement, a violent explosion takes place. This is accompanied by evolution of much heat and gases

V.A. Belakon, who studied at Physico-Technical Institute, proposed the theoretical explanation of the phenomenon described by

Pokrovskii and in abstract of Maj Kudravetz

The particles of gases formed during a lightning ball expln, as well as in ordinary explns, carry considerable electric charges. In the expansion of these gases, the fluxes of their particles move, as a rule, irregularly. Streams of gases carrying one charge may fly considerably forward, while mass of gases with opposite charge may lag behind. This results in the appearance of the electromagnetic field alternating in space and in the emission to all sides of a powerful, but very short-lived, electromagnetic impulse

Explosion, Polytropic Curve and Polytropic Law. See Vol 4, pp D474-R & D475-L

Explosion of Powdery Explosive Charges. See Vol 4, pp D475-L to D476-L

Explosion Power or Strength. See Vol 4, pp D476-L to D479-R

EXPLOSION, PREMATURE

Premature Explosions of Projectiles (Éclatements prématurés des projectiles – in French) (In collaboration with N.D. Baron of ADED, Picatinny Arsenal).

Premature explosions of projectiles inside a cannon may be due to many causes but, due to the fact that anything tangible bearing on the cause is blown to pieces, an investigation is very difficult. It is very rare for anything to be found among the exploded fragments of the projectile or gun that will throw definite light on the probable cause of a premature. Consequently, when the cause of a particular premature explosion is investigated, it is usually necessary to consider all the different possible causes and then from the evidence at hand to deduce the most probable cause in the case

According to Major C.M. Steese, Ordnance Officer at Fort Sill, USA, in any particular projectile there are about 36 distinct and different components and it is possible for any one of them to cause a premature

Following is a list of some of the causes of prematures;

1) Some defect in the **barrel of a cannon** which would cause slowing down of the projectile or cause a sudden increase in the speed of it. For instance, if the rifling of the barrel is damaged or is not smooth in spots, the projectile might suddenly stop or slow down and if the resulting impact is sufficiently great, the bursting charge will explode. If the fuze is armed by inertia, detonation will be complete, but if the fuze is not armed the detonation will be incomplete. More accidents result from the use of new guns than from older ones and more from howitzers than from guns

Note A. The reason new guns might be the cause of more accidents than ones slightly used may be explained by the fact that the bore of new guns is not very smooth

Note B. One of the reasons why there are more accidents with howitzers than with guns is that howitzers use faster propellants. This means that the initial speed of burning is greater and the maximum pressure is reached in a much shorter period. If there is a defect in a barrel near the breech, then the impact will be greater in the case of a howitzer than in the case of a gun

If a barrel had been damaged previously by a small incomplete detonation, it would have one or several sections with enlarged diameter. When a projectile hits such a spot, it becomes disengaged from the rifling and suddenly gains speed. If the charge does not explode from this, it is sure to when the projectile hits the rifling of the narrow section of the barrel

Defects in the metal of a barrel, such as cavities etc, might cause the bursting of a cannon by pressure of the propellant gases, even though the projectile did not explode

2) Some defects in the **construction of a projectile**, such as weak metal, presence of flaws, cracks etc. For instance, if the steel had not been properly annealed, the projectile might break or crack inside the barrel and the propellant gases would ignite the bursting charge. The charge might also be ignited if the tail fuze were not properly assembled

3) Some defects in the **bursting charge of a projectile**, such as cavitation, exudation etc. If a cavity is in the rear section of a projectile, the firing causes "setback" of the charge. If the cavity is in the forward section, there is danger that the shell will not explode even on striking the target because the fuze and booster might become separated from the bursting charge

4) Some defect in construction of the **fuze or of the booster** might cause a premature but such cases are comparatively rare. If the fuze or booster detonates it causes complete detonation of the bursting charge

5) If the **cartridge case is too short**, in the case of fixed ammunition, then the projectile does not reach the rifling. When the propellant is ignited, the gases push the projectile forward very rapidly and as soon as it reaches the rifling, a momentary slow down or a complete stop might take place. As result of this sudden change in velocity, a premature might occur

6) If the **propellant is very fast burning**, the push on the projectile might be so sudden and great that it might cause a premature

7) If the propellant is very **irregularly burning**, which might be caused by nonhomogeneity, the the projectile will move in a jerky fashion, which might result in a premature

Note. According to Refs 1 & 2, more prematures occur with guns of larger than caliber 75mm than with those of 75mm or below. No explanation of this is given

8) Worn gun tubes may be responsible for premature functioning of shells in that the fuze may be armed and activated by the erratic movement of the projectile in the weapon. The erratic movement of the projectile is caused by the worn rifling not properly engraving the rotating band of the shell

9) Some American shells loaded with 50/50 Pentolite prematured and it was believed that the cause was exudation thru the closure at the base of the shell and that the exudate set up an explosive train which was subsequently ignited by the propellant when the gun was fired. When similar shells were later tested, it was shown that heating above the melting point of Pentolite (170°F) caused exudation and leakage of the exudate thru the closure at the base of the shell. Leakage also occurred at the forward end of the projectile thru the crimp between the hemispherical liner and shell body into the cavity beneath the fuze. When similar shells were subjected to a "cycling treatment", alternating 16 hours at 160° F and 8 hours at −65°F, exudate leaked from some shells, while in others a gummy material exuded from the base plug within 3 days. On storing at 149°F for 66 days, no exudation occurred, but the Tetryl pellet of the booster became cemented to the bursting charge by material which exuded from the Pentolite (Ref 4)

Refs: 1) A. Niblach, Field Artillery Journal 1928 (March-April), pp 145–50 (Prematures in the USA) 2) E. Dunin-Marcinkiewicz, MémArtilFr **14**, 155–74 (1935) (Sur les éclatements dans l'âme des bouches à feu); Translated from PrzegladArtyleryjski **1930**, No 4 3) G.L. Schuyler, US Naval Report, Oct 21, 1946, "The Diagnosis of Puzzling and Important Misbehaviors of Naval Ordnance Material". The following prematures and their possible causes are cited: a) **The Colorado's 5" 25 caliber gun explosion.** An AA projectile gave a bore premature which burst a gun killing most of the crew. About 6000 similar projectiles were carefully examined for defects but nothing suspicious could be found. About ten years later it was found that the base of some of these projectiles contained tiny perforations and it is possible that hot propellant gases penetrated inside the shell when it was fired. When shells with artificially perforated bases were fired experimentally, prematures were obtained

b) **40mm loading accidents.** Occasional premature explosions took place when 40mm shells were "catapulted" into the guns by the loading mechanisms and some personnel were killed and injured by the blowbacks. Extensive investigation revealed that the primer mixture occasionally broke up from shocks and vibrations in handling and shipment and sifted out in such a way as to render the primers supersensitive

c) **Bore prematures due to loose or missing tracers.** During WWII, there were a very small number of bore prematures among the enormous number of 5" AA rounds that were fired. As no cause could be determined, the fuze was replaced by another type but an occasional premature still occurred. Only after 4 years of extensive investigation was it found that prematures were not caused by faulty fuzes but by insecurely attached tracers. Experimental firings showed that if a tracer becomes loose and is detached, the propellant gases might enter the cavity from the tracer and then break thru the wall of the fuze, thus reaching the filler

4) Aberdeen Proving Ground, Firing Record No P-4453, Jan 10, 1949 5) Ballistic Research Laboratory Report No 452 by L. Zernow and O.P. Bruno, "Study of Prematures with M48 and M51 Fuzes" 6) Aberdeen Proving Ground, "Investigation of 20mm High-Chamber Pressure Malfunctions", 15th Report on Project TS3-3104. Firing Record S-43,260 (1951) 7) Following is a list of Picatinny Arsenal Technical Reports on Premature Explosions of Projectiles or Investigations of the Causes:

a) G.E. Rogers, "Premature of Round, Shell HE Mk II in 37mm Model 1916", PATR **207** (April 1932)

b) V.R. Reed, "Investigation of the Cause of Prematures in Ballistic Tests of M39A2 Point Detonating Fuzes", PATR **698** (May 1936)

c) C.J. Bain, "Premature of 155mm HE Shell, Mk III" PATR **805** (March 1937)

d) D.R. Beeman, "Investigation to Determine Cause of Prematures in Firing 37mm Ammunition, Lot 1075-22 at Fort Lewis", PATR **883** (Feb 1938)

e) D.R. Beeman, "Investigation of Premature of 37mm Ammunition, Lot PEM 12536-1 at Pueblo, Colorado", PATR **920** (Sept 1938)

f) D.R. Beeman, "Premature Functioning of 37mm T2 HE Shell, Cast TNT Loaded with T30 PD Fuze, Lot E621-1", PATR **939** (Dec 1938)

g) D.R. Cameron, "Examination of 1000 3" AA Shell from Ammunition Lot 2724-6", PATR **1099** (July 1941)

h) R.W. Scharf, "Examination of 1138 3" AA Shell M42 from Ammunition Lot 2724-6", PATR **1109** (Aug 1941)

i) D.R. Cameron, "Examination of 1000 3" AA Shell from Ammunition Lot 2724-6", PATR **1112** (Aug 1941)

j) R.W. Scharf, "Examination and Loading of 3" AA M42 Shell with Defective Bases, Lot 2292", PATR **1120** (Oct 1941)

k) F.H. Vogel, "Examination of 155mm Shell, Lot 1-7993-22, Received from Wolf Creek Ordnance Plant", PATR **1173** (June 1942)

l) F.H. Vogel, "Examination of 75mm Mk 1 Shell, Lot NOD 5340-2, Received from the Nansemond Ordnance Depot", PATR **1199** (Sept 1942)

m) V.R. Reed, "37mm Mk II Practice Shell-Investigation of Premature Functioning", PATR **1244** (Feb 1943)

n) F.H. Vogel, "Examination of 10 Each 155mm Mk 1 HE Loaded Shell, Lot DOD-25112-13", PATR **1373** (Dec 1943)

o) F.R. Benson, "Premature Explosions of 150-lb GP T1 Bombs", PATR **1537** (June 1945)

p) A.L. Dorfman, "Investigation of Malfunctions with T43-T47 Colored Smoke Aircraft Signals", PATR **1573** (Dec 1945)

q) L. Jablansky, "Investigation of Segregation in Composition B", PATR **2019** (April 1954)

r) G. Demitrack, "Investigation of Malfunctioning of 4.2 Inch Mortar Ammunition", PATR **2049** (Aug 1954)

s) L. Jablansky, "Heat Study of Composition B-Loaded 155mm M101 HE Shell", PATR **2103** (Dec 1954)

t) S.J. Lowell, "Investigation of Prematures in 75mm T50E2 Shell", PATR **2104** (Jan 1955)

u) I.S. Kintish, "Effect of a Hot Weapon on Composition B-Loaded 105mm M1 HE Shell", PATR **2131** (Jan 1955)

w) J. Davis, "Unsteady Temperature Distribution in a Shell Wall", PATR **2140** (Feb 1955)

x) S.J. Lowell, "Study of Grit and Exudate as Possible Causes of 75mm T50E2 Shell Prematures", PATR **2168** (April 1955)

y) L. Jablansky, "Laboratory Scale Test Device to Determine Sensitivity of Explosives to Initiation by Setback Pressure", PATR **2235** (Sept 1955)

z) S.J. Lowell, "Firing Safety of Production Lots of Composition B-Loaded 75mm T50E2 HE Shell", PATR **2236** (Sept 1955)

aa) S.J. Lowell & E.A. Skettini, "Investigation of the Safety of Firing Composition B-Loaded 155mm M101 HE Shell", PATR **2329** (May 1956)
bb) S.D. Stein & S.J. Lowell, "Study of Exudation as a Possible Cause of 75mm T50E2 Shell Prematures", PATR **2344** (Aug 1956)
cc) S.D. Stein & S.J. Lowell, "Investigation of Prematures in 75mm T165E11 HEP-T Shell", PATR **2359** (July 1956)
dd) L. Jablansky, "Factors Affecting Sensitivity of Composition B to Setback", PATR **2433** (June 1957)
ee) S.D. Stein & R.G. Salamon, "Investigation of a Premature of a Composition B-Loaded 120mm T15E3 HE-T Shell", PATR **2507** (Jan 1958)
ff) S.D. Stein & M.J. Margolin, "Proposed Shell Loading Standards Based on a Statistical Study of Setback Sensitivities", PATR **2563** (Nov 1958)
gg) T.W. Stevens, "Investigation of Prematures and Deflagrations of 30mm T306E10 and T306E11 Shell", PATR **2565** (Oct 1958)
hh) S.D. Stein, "Investigation of a Premature of a Composition B-Loaded 240mm M114 Shell", PATR **2573** (Nov 1958)

Premature Firing of Rounds, or Premature Ignition of Propellants (In collaboration with N.D. Baron of ADED Picatinny Arsenal)

It sometimes happens that when bags of propellant of separate loaded ammunition are charged into a gun immediately after the previous round had been fired, a premature ignition of the charge takes place. This usually happens when one or several pieces of an incompletely burned powder bag from the previous charge remain glowing in the barrel. It has been claimed that cotton bags are usually the cause of such accidents and for that reason, powder bags in this country have usually been made of silk, now partly replaced by Nylon. On the other hand, the Russians have always used cotton bags either unmercerized or 50% mercerized (the threads running in one direction are mercerized while those running in a perpendicular direction are unmercerized). During WWII, the Germans used cylindrical casings made of double-base powder instead of bags. Such casings burned just as completely as the propellant

In firings involving a high rate of fire, particularly with machine guns and anti-aircraft guns, automatic functioning or **"cook-off"** of a round left in the hot gun for a period of time is possible. This "cook-off" condition may result in either spontaneous ignition of the primer or the propellant powder

Refs: 1) Blinov, Course of Artillery, Moscow (1948–1949) 2) US Ordnance Safety Manual, **ORD M 7–224**, Revised 12 August 1952, Section 2818 3) US Army Materiel Command Regulation, "Safety Manual", AMCR **385–100**, April 1970, p 28–16 (Cook-Off) & 28–17 (Premature Bursts)

Premature Explosions in Shooting Wells. In drilling holes thru certain shales, particularly those associated with oil and gas, the presence of areas which contained very finely divided pyrite was observed. When such areas are exposed to the atmosphere (especially if it is moist and warm), oxidation of the pyrite to the RSO_4 form takes place. As this is usually accompanied by considerable evolution of heat, there is danger of a premature explosion if a blasting charge is introduced into the hole in order to shoot a well

Refs: 1) D.F. MacDonald, USBureauMines Bull **86**(1915) (Describes premature explosions in the Canal Zone) 2) D.F. MacDonald, OilTrade J **11**, 114–5(1920) & CA **14**, 2713(1920) (Probable cause of premature explosion in shooting wells)

Explosion Pressures and Their Measurements. See Vol **4**, pp D483 to D491-R and AddnlRef: E.O. Genzsch, "Explosionsdruck als formgebendes Mittel" (Explosion Pressure as Formshaping Means), Explosivstoffe **1964**, 89–93 & 105–08

Explosion by Priming or Boostering. See Vol 4, p D494-L

Explosion Process, The Hypothetical Rate in the. A discussion of the hypothesis that a type of radiation plays a predominant role in the mechanism of the process of detonation of expl solids and liquids and in the burning of proplnts
Ref: E. Sanger, IndChimBelge **20**, 289–92 (1955) & CA **50**, 17454(1956)

Explosion, Products of. See Vol 4, pp D494-R & D495-L

Explosion Propagation Thru Explosive Charges. See Vol 4, pp D497-L to D498-R

Explosion Propagation Thru Layers of Non-explosive Substances. See Vol 4, p D498-R

Explosion Properties and Blasting Action of High Explosives. Title of Chapter 11 in Cook's book (1958), pp 265 to 282 (9 refs)

Explosion, Protection in. A review of approved electric switches, motors, meters, recorders and other gear suitable for use in areas where explosives are handled. Being spark proof, they minimize explosion hazards
Ref: H. Busch, "Die Schutzarten im Explosionsschutz" (Types of Protection in Explosion Protection), Explosivstoffe **1958**, 54–9 (9 refs)

Explosion Pulping. In this process, wood chips about ¾ inch long are fed into a battery of digesters (called guns), in which the chips are steamed gradually to a pressure of 1100 to 1200 psi and then, after holding at that pressure for about 5 secs, are released by means of a quick-opening valve into a cyclone. The explosive effect of this rapid release breaks up the wood chips into a fibrous mass

This treatment does not remove the lignin. The fibers are dark in color and very stiff. They are not used in paper manuf nor for the nitration of NC, but can be pressed into a product of extreme durability & hardness, known as "Masonite Presswood"
Ref: J.P. Casey, "Pulp and Paper", Interscience, NY (1949), pp 205–06

Explosion, Rarefaction Effect in Air Blast. See Vol 2, p B180-R

Explosion, Rarefaction Wave and Release Wave of E.M. Pugh. See Vol 4, pp D500-R to D502-L

Explosion Reactions; Physico-Chemical Investigations of. Heats of explosion of various chemicals were calculated from thermodynamic considerations and obtained experimentally in bomb tests
Ref: R. Dautel, Physikalisch-Chemische Untersuchung von Explosions Reaktionen, Explosivstoffe **1963**, 10–18 (14 refs)

Explosion Reactions During Reduction of Fluorinated Compounds with $LiAlH_4$. Extreme caution should be used in reducing any fluorinated compd with $LiAlH_4$. Violent reactions are described involving 2 dif fluoro compds. Suggestions for safe operation include treating small amts of $LiAlH_4$ in ether with a small quantity of compd to be reduced, with drying and slow heating or carrying a trial reaction
Ref: W. Karo, C&EN **33**, 1368(1955) & CA **49**, 9278(1955)

Explosions; Relation Between Hardness, Melting Point and Sensitivity. Initiation by friction and impact of PETN is studied in the presence of the following grits: NH_4NO_3, $KHSO_4$, $AgNO_3$, $Na_2Cr_2O_7$, NaOAc, KNO_3,

$K_2Cr_2O_7$, AgBr, $PbCl_2$, AgI, Borax, bismuthinite, glass, rock salt, chalcocite, galena, and calcite. No obvious correlation between sensitivity and the hardness or chem properties of the grit was observed. However, if it is compared to the mp, a sharp division is apparent. All grits with a mp greater than 430° are effective in causing expln, while all grits of mp less than 400° are ineffective. Other secondary explosives, whether solid (Cyclonite) or liquid (Nitroglycerine) behaved in a similar manner. Primary explosives (Lead Azide, Lead Styphnate etc) also gave similar results, but limiting mp of the grit varied with the nature of explosive and was somewhat higher (500 to 550°). Decreasing size of grit below a few microns diminished its sensitizing effect. Particles of SiC or diamond as small as 5×10^{-5} cm in diam still have an appreciable sensitizing effect
Ref: F.P. Bowden, ProcRoySoc(London) **A204**, 20–5(1950) & CA **45**, 10588(1951)

Explosion, Resistance to. See Vol **4**, pp D508-L to D510-L

Explosion Retardation. The sample-size effects of the explosion retardation of mercury and silver oxalates and fulminates are discussed
Ref: V.V. Boldyrev, ZhurFizKhim **30**, 1088–91(1956) & CA **50**, 17454(1956)

Explosions, Review. A review of information on selected aspects of gas and vapor explosions are given by K.N. Palmer, JInstFuel **29**, 293–309(1956) & CA **50**, 12481 (1956)

Explosion of S-IV All Systems Vehicles, Investigation of is described by J.B. Gayle of the National Aeronautics and Space Administration, in Technical Note **TND–563**, Sept 1964

Explosion of Shells and Bombs. Generally, a shell or a bomb filled with an HE (high explosive) detonates if properly initiated, leaving no residual explosive behind. The color of the smoke produced is either white, grey or black, depending on whether or not the explosive filling contains sufficient oxygen for complete combustion. If, however, the shell (or bomb) is not properly initiated, or if there is some defect in its construction or in the composition of the explosive itself, the entire charge is not consumed and the shell (or bomb) is said to "explode" instead of detonate. This is revealed by the color of the smoke which would be yellowish or brownish, more or less of the color of the explosive itself, because the smoke would contain some unconsumed (residual) explosive
Refs: 1) Marshall **2**(1917), 550 2) C.G. Dunkle, Syllabus, "Detonation Phenomena", Picatinny Arsenal-Stevens Graduate School Program, Issue 1958, pp 325–28 (Explosion of Shells and Bombs)

Explosion, Shock Wave in. See Vol **4**, pp D530 to D542-L

Explosions in Space (Raumexplosionen, in Ger) are described in Refs
Refs: 1) H.H. Freytag, "Raumexplosionen durch elektrische Anlagen", VerlagChemie, Zürich (1949) 2) H.H. Freytag, "Handbuch der Raumexplosionen", VerlagChemie, Weinheim/Bergstr (1965), 664 pp

Explosion Spectra of Metals. Anderson was the first to use the "explosion" method of exciting spectral lines of a metal. He studied the flash of light emitted when a thin wire of a metal to be studied was exploded by an oscillatory discharge from a condenser charged to several thousand volts. As the atoms of the metal are subjected to a powerful stimulus very abruptly, the conditions of excitation are very much different from those prevailing in an arc of the same metal carrying a low current. Several investigators have developed this tech-

nique and have studied the explosion spectra of a number of substances

Sawyer and Becher (Ref 2), Smith (Ref 3), Nagoaka and Futagami (Ref 4) modified the method and accumulated some data

Venkatesha of India (Refs 5 & 6) constructed a special apparatus and studied the difference between the intensities of spectral lines excited in the fuse spectra of Ag, Al, Mg, Ti and in the corresponding low current arcs. The apparatus consisted of a Hilger E2 quartz spectrograph, an electric bulb with a straight vertical filament and a wire or thin strip of the metal under investigation, placed between two electrodes 4cm apart, made from the same metal. The wire was interposed between the slit of the spectrograph and the lamp so that illumination from the lamp is cut out. The shadow of the wire is focused on the slit by a quartz lens of short focal length. The electrodes of the wire were connected thru a double pole switch to the leads from a battery of storage cells of total voltage 120 volts. When the switch was closed, the wire fused, emitting an intense light for a fraction of a second

These investigators showed that more lines are shown in explosion spectra than in the arc spectra. The weak lines of arc spectra, especially those due to impurities, are shown much brighter by the new method. In many cases, traces of impurities undetectable by the arc spectrum were easily seen in the explosion spectra. The continuous spectrum was, as a rule, stronger than that obtained by the arc method

Refs: 1) J.A. Anderson, AstrophysJ **51**, 37 (1920) 2) R.A. Sawyer & A.L. Becher, Ibid, **57**, 98(1923) 3) S. Smith, Ibid **61**, 186 (1925) 4) N. Nagoaka & T. Futagami, InstPhysChemResearch, Tokyo; SciPapers **530**, 111(1934) and Imp-AcadTokyoProc **11**, 43(1934) 5) S. Venkatesha, Current Science (India) **10**, 287(1941) 6) S. Venkatesha, JMysoreUniv(India), Sect B, **2**, 55–60 (1941)

Explosion Spectra and Spectrographic Measurements. See Vol **4**, pp D548-R & D549-L

Explosion (and Combustion) Spherical. See Vol **4**, pp D549-L to D556-R

Explosion Spherical; Initial Behavior of. Disturbance near source of spherical blast from a polytropic expl in a surrounding medium is analyzed. A second blast wave is the result of the breakdown of continuous gas flow in the neighborhood of a singular characteristic. For all types of expls, the 2nd shock is a 2nd order effect in terms of the square root of the time from the end of detonation. Application to PETN charges in air and water is discussed

Ref: M. Holt, ProcRoySoc(London) **A234**, 89–109(1956) & CA **50**, 6793(1956)

Explosion, Spontaneous. See Vol **4**, pp D561-R to D563-R

Explosion, Stability and Instability of. See Vol **4**, pp D569-L to D574-R

Explosion-State Products of Dynamites, TNT, LOX and AN Explosives. The explosive properties of various commercial and military expls are given in tabular form. The expls include TNT, Dynamite, high-AN-NON-NG, LOX and water compatible types. Such properties as heat capacity $\overline{C}_v$, heat of explosion Q, absolute temp T, pressure p, and number of molecules/kg are given

Ref: Cook (1958), 306–21

Explosions by Static Electricity Initiated, Demonstration of. Apparatus is described for initiating an explosion in an Et_2O–air–O mixture by static electricity

Ref: D.S. Ainslie, AmerJPhys **24**, 408(1956) & CA **50**, 13443(1956)

Explosion, Suppression System. Like any other protection system, an explosion suppression system is made up of three com-

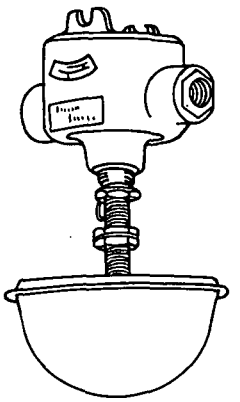


Fig Ex1

ponents: devices to (1) sense the explosion, (2) translate the signal into action, and (3) prevent the reaction from continuing. The system shown is comprised of a sensing detector (top) electrically coupled to an explosively actuated extinguisher. The explosion detector, sensitive either to light or pressure, senses ignition within the protected volume before the flame can propagate more than a few inches. This action completes the electrical circuit from the power supply to the suppressing device (bottom)

This particular device consists of a frangible hemispherical container, filled with an inerting agent and having an EED at its center. The initiator, when set off by the sensing signal, fires and ruptures the hemisphere along the score marks. Thus the inerting agent is dispersed rapidly. Elapsed time from detection of an incipient explosion to suppression of the mixture may be only a few milliseconds. Typically, suppressed explosions in practical applications will peak at a pressure anywhere from one to three psi over a maximum time of 150 milliseconds. Company makes line of electric temperature controls, detection, and indicating devices *Refs:* 1) Fenwal, Inc, 251 Main St, Ashland, Mass 01721 2) Gunther Cohn, Edit, Expls&-Pyrots **3**(11)(1970)

Explosion, Sympathetic or Explosion by Influence See Vol **4**, pp D395-R to D402-L

Explosion (and Detonation) Temperature and Its Determination. See Vol **4**, pp D583-L to D589-L and also Ignition (or Explosion) Temperature Test in Vol **1**, p XVI

Explosion Temperature Determination as Conducted at Picatinny Arsenal. The method described by A.J. Clear in PATR **3278**(1965), pp 7–8 with Fig 8 on p 39 is also described in Vol **4** of Encycl, pp D583 & D584

Explosion Temperature Determination by Rumpff's Method. Automatic apparatus for detn of expln and deflagration temps of explosives and propellants is described by H. Rumpff, Explosivstoffe **1957**, 113–14

Explosion, Temperature Developed on. See Vol **4**, pp D589-L to D601-L

Explosion, Temperature Developed on. Calculation for Permissible Explosives. See Vol **3**, pp C445-R to C449-R

Explosion, Temperature Developed on. The relation between the temperature of explosion of a propellant and the speed of combustion at rocket pressures was discussed by P. Tavernier & C. Napoly in MP **37**, 331(1955)

Explosion (and Explosives) Terminology. Explosion has several attributes and hence can be described or defined in different ways. From the standpoint of chemistry, it is a rapid chemical process resulting in the evolution of gas and heat. To the classic physical definition of a high-pressure energy release must be added thermonuclear effects. Both chemical and physical concepts must be combined to obtain a complete terminology *Ref:* H. Pessiak, Explosivstoffe, **1960**, 23–6, 45–7

EXPLOSION THEORIES. See under DETONATION (AND EXPLOSION) THEORIES in Vol **4**, pp D601-L to D602-R. See also "Theory of Explosion Processes", N.S. Akulov, DokladyAkadNaukSSSR **104**, 376–9 (1955) & CA **50**, 9739 (1956)

Explosion Theories for Gases. Recent developments of explosion theories were reviewed by Gray & Yang (Ref 3). The models of thermal and isothermal chain theories of explosion are mathematical idealizations constructed to characterize laboratory observations. They have yielded useful understanding of the explosion phenomenon in cases when physical conditions are consistent with these idealizations. Unfortunately, the attitude of regarding both theories as convenient models useful in particular instances has often been replaced by the outlook that "real" explosions are either chain or thermal, thus giving rise to the setting up of criteria to decide to which class a given case belongs. For example, Semenov (Ref 1) stated: "A thermal explosion must be preceded by a slow but perfectly measurable reaction. A chain explosion takes place under conditions of immeasurably slow reaction. In a thermal explosion the heat liberated by the reaction is the *cause* of the explosion. In a chain explosion, the evolution of heat is a *consequence* of the chain avalanche"

While Gray & Yang are not denying the usefulness of such ideas, they consider that too literal an application on the distinction can lead to difficulties. For this reason they tried to unify both theories and this problem is discussed in their paper. They also examined the effect of fuel consumption on thermal explosions, definition of critical conditions and the effects of vessel shapes. Finally, the relationship between thermal explosion criteria and flame theory described by Belles (Ref 2), as well as detonability limits were pointed out. Comments on the paper of Gray & Yang of Profs R.R. Baldwin & R. Ben-Aim are given on p 1061 of Ref 3

Refs: 1) N.N. Semenov, "Chemical Kinetics and Chain Reactions", OxfordUnivPress, Oxford (1938); Vol 2, "Some Problems of Chemical Kinetics and Reactivity", Pergamon Press, NY (1959) 2) F.E. Belles, 7thSympCombstn (1959), 745 3) B.F. Gray & C.H. Yang, "The Present Theoretical Position in Explosion Theory", 11thSympCombstn (1967), 1057–61 (18 refs)

Explosion (Detonation, Deflagration and Decomposition), Thermal Theories and Thermochemistry of. See Vol **4**, pp D619-L to D622-L

Addnl Refs on Thermal Explosions: A) D.M. Clemmow and J.D. Huffington, TransFaradSoc **52**, 385–96 (1956) & CA **50**, 14229 (1956) (Extension of the theory of thermal explosion and its application to the oscillatory burning of explosives) B) F. Kaufman & N.J. Gerri, "Experimental Studies of Thermal Explosions and of Moderately Fast Reactions", 8thSympCombstn (1960) (Pubd 1962), pp 619–27 C) D. Gross & A.B. Amster, "Thermal Explosions: Adiabatic Self-Heating of Explosives and Propellants", Ibid, pp 728–34 D) F.P. Bowden et al, "The Thermal Decomposition of Explosive Crystals (A Study by Electron Microscopy and Diffraction)", Ibid, pp 836–42 E) Z.G. Szabó & J. Száva, "Factors Which Influence the Thermal Decomposition of Potassium Picrate", Ibid, pp 863–72 F) B.J. Tyler & T.A.B. Wesley, "Numerical Calculations of the Critical Conditions in Thermal Explosion Theory with Reactant Consumption", 11thSympCombstn (1967), pp1115–21 G) P. Gray & P.R. Lee, "Studies on Criticality: Temperature Profiles in Explosive Systems and Criteria for Criticality in Thermal Explosions", Ibid, pp 1123–31

Explosions of Ti in HNO_3 and Zr in HNO_3. Reports are given of explosions occurring when Ti test specimens were used in red fuming nitric acid. The cause of such explosions is unknown. The use of Zr in nitric acid solutions is described briefly

Ref: J.A. English et al, Corrosion **12**, 658 (1956) & CA **50**, 9988 (1956)

Explosion-Time Test of Double-Base Propellants. It was concluded from a large number of tests that the expln test does not measure the true stability of smokeless proplnts contg NG. Some proplnts rendered unsuitable by long heating at 78° still gave satisfactory expln tests at 134.5°. Addn of as little as 0:10% K nitrate caused a distinct lowering of expln time, but did not affect other tests. The presence of a stabilizer had no effect on the expln test. The effect of addg various inorg salts (from 1 to 4%) on proplnts contg NG 40, NC(13.08%N) 50.25 & DPhA 0.75% was also reported
Ref: D.R. Wiggam & E.S. Goodyear, IEC (AnalEd) **4**, 77–8(1932) & CA **26**, 1444(1932)

Explosion of TNT Plant at Saint Chamas, France. A detailed description and analysis of the causes of an expln on 16 Nov 1936 which caused the death of 53 persons are reported. Pb & Fe were found to produce expl substances from TNT in the presence of nitric acid which could be ignited by heat, shock, friction, or contact with concd nitric & sulfuric acids. Salts of Pb & Fe were found at the scene of the expln
Ref: A. Kovache & H. Thibon, MP **34**, 369–88(1952) & CA **49**, 4293(1955)

Explosion of Trinitronaphthalene Plant at Saint Chamas, France. On 4 April 1940 during manuf of Trinitronaphthalene, an expln occurred in the No 8 nitrator causing the death of 11 persons and injuring 53 others. The bldg was severely damaged. It was built in 1915 for making TNT but was transformed in 1924–25 for making Nitronaphthalenes. The buildg contained 12 nitrators of the Düsseldorf type arranged in a line. The capacity of each nitrator was 4 cubic meters, all equipped with stirrers operating at 100rpm

During the manufg process, the operator observed a normal temp, but the grains of Trinitronaphthalene became large and the temp reached 60°. Due to the formation of large agglomerates of crystals, the belt broke and the stirrer became inoperative. The lack of agitation prevented cooling of the nitrator and local heating (80°) took place accelerating the oxidation process. All of these factors prevented the nitrator from being discharged in the usual manner and contributed to the cause of explosion
Ref: Anon, MémPoudres **34**, 389–95(1952) & CA **49**, 4293(1955)

Explosions, Unexpected. *$POCl_3$ Explodes in Lab.* A severe and unexplained expln of a bottle contg recovered phosphorus oxychloride occurred at the Univ of Arizona chem lab. Dr L. Plummer was prepg the diacid chloride of ferrocene-1,1-dicarboxylic acid, using a mixt of PCl_5 & phosphorus oxychloride. He had just distilled (under reduced press) the oxychloride from the reaction mixt. The recovered phosphorus oxychloride was poured into a 500-ml brown bottle contg some of the compd recovered from earlier, similar reactions. Some of the recovered compd had been standing for ca 3 months

As the bottle was placed on a desk, an expln took place; the blast was forceful and was accompanied by a sharp report. Glass splinters from the bottle severely cut Dr Plummer and the $POCl_3$ caused severe burns

No satisfactory explanation of this expln has been found, reported Dr C. Marvel, nor was he aware of similar incidents involving $POCl_3$ (Ref 1)

Explosion at US Industrial Chemical Plant. An expln at the US IC, Tuscola, Ill plant which killed one man and injured 13 others was reported. Cause of the expln, which occurred in the plant's ammonia synthesis area, was not determined. Preliminary investigation indicated the blast resulted from ignition of hydrogen gas escaping from a ruptured pipe. The control room & one compressor buildg wall were severely damaged (Ref 2)

Methyl Ethyl Ketone. Very little was left of a truck after its load of Methyl Ethyl Ketone exploded in Bromwich, near Birmingham, England. Thirty persons in nearby

houses were injured, but no one was killed. The driver had noticed smoke, parked in a vacant lot, and was away making a telephone call at the time of the blast (Ref 3)

Explosion at Firestone Tire. An expln at Firestone Tire's Akron, Ohio research center killed veteran chemist Christian E. Best and injured 13 others. Damage was estimated at 1 million dollars. The blast occurred in a second-floor plastics lab and may have been caused by ignition of propylene fumes from a leaking cylinder. What ignited the volatile propylene fumes was not determined (Ref 4)

Explosion at Olin Mathieson. A blast in the propylene oxide-ethylene glycol area of Olin Mathieson's Brandenburg, Ky org chemicals plant injured 29 persons and caused an estimated 8 million dollars damage in April 1962. Olin believed that a pump relief valve failed and allowed ammonia to mix with ethylene oxide, causing a tank expln (Refs 5 & 9)

Explosion at Pittsburgh Metal Purifying Company. An expln & fire leveled the plant and offices of Pittsburgh Metal Purifying Co, Mars, Pa killing 2 workers & injuring a third worker. The firm estimated damage at ca 1 million dollars. The company makes exothermic materials used in foundries & steel mills (Ref 5)

Sinclair Oil Depot. Expln & fire struck the Sinclair Oil depot along the Brooklyn waterfront on 10 May 1962. The fire, which was finally brought under control after 3 hrs, sent dense black smoke billowing skyward. No injuries were reported (Ref 6)

Explosion from Chlorinated Rubber-Zinc Oxide Reaction. An until-now undescribed exothermic reaction – that of chlorinated rubber with zinc oxide was responsible for an expln that leveled the manufg area of Dayton Chemical Products Laboratories, West Alexandria, Ohio in April 1962. The blast took place during mixing of a large batch of material including the two reactive ingredients along with other materials. Personal injuries involved only first & second degree burns which reqd only brief treatment (Ref 7)

Olin Mathieson Plant Explosion. The hydrazine unit at the company's Lake Charles, La chemical plant was extensively damaged by an expln 6 October 1962. One operator was killed and 4 other men were injured. Damage was estimated at over 1 million dollars (Ref 8)

NOTE: The above described Unexpected Explosions occurred in one year arbitrarily selected as 1962. No attempt was made to cover the extensive literature on this subject covering any other period of time

See also Explosions, Uninvited in this Vol

Refs: 1) Anon, C&EN **40**, No 3, p 55 (15 Jan 1962) 2) Anon, C&EN **40**, No 4, p 21 (22 Jan 1962) 3) Anon, C&EN **40**, No 9, p 110 (26 Feb 1962) 4) Anon, C&EN **40**, No 14, p 21 (2 April 1962) 5) Anon, C&EN **40**, No 17, p 21 (23 April 1962); **40**, No 18, p 124 (30 April 1962) 6) Anon, C&EN **40**, No 21, p 145 (21 May 1962) 7) Anon, C&EN **40**, No 37, pp 79–80 (10 Sept 1962) 8) Anon, C&EN **40**, No 42, p 21 (15 Oct 1962) 9) Anon, C&EN **40**, No 53, p 75 (31 Dec 1962)

Explosion, Underwater. See Vol 4, p D628-L to D629-L

Explosions, Uninvited. Under the title of "Uninvited Chemical Explosions", warning was given by the late Mr Tomlinson & Dr Audrieth (Ref 3) about hazards in preparation and handling of substances which were never previously considered as being explosive, but recently proved to be such. Among them were cited many oxygenated metal ammine complexes such as Hexammine chromium (III) nitrate $[Cr(NH_3)_6](NO_2)_3$ and Hexammine cobalt (III) perchlorate $[Co(NH_3)_6](ClO_4)_3$, each nearly as powerful an expl as TNT (Ref 1). (More than seventy ammine complexes are described in Vol 1 of Encycl, pp A277 to A282). Many other dangerous, but previously not considered as such compds

and mixtures are listed in the Refs. If dangerous properties of substances prepd and handled in the laboratory and in the plant would be known in advance, many accidents could be avoided by taking necessary precautions and obeying safety rules

A compound or a mixture containing an appreciable amount of oxygen, along with a significant quantity of nitrogen, should be suspected, and it would be foolhardy to ignore the potential danger of any substance whose elemental components could undergo exothermic recombination with formation of very stable products. The simplest and most accurate way to evaluate the potential hazard of any product is to calculate its heat of decomposition to the most stable products. This can be done by making use of the Pauling bond energies and the heat of formation of the decomposition products

Lothrop & Handrick (Ref 2) have shown that there is a definite relationship betw heat of decomposition and oxygen balance and have indicated a simple method for calculating the former. The oxygen balance concept can be used, *a priori* to good advantage, since expl power reaches a maximum when OB to CO_2 & H_2O is near zero. It is those materials, contg just sufficient oxygen in the molecules to form H_2O, CO_2, and N_2, that are capable of releasing the largest quantities of energy per gram and will, in general, be most destructive

The recombinztion of atoms from a relatively weakly bound state to more stable states may also occur where two or more components are brought together in proper proportions, and where the energy of reaction is such that its release is effected with great rapidity, especially if large volumes of gaseous products are formed. Thus an association of oxidant and reductant can be just as dangerous as a single expl compd. In the case of mixtures, however, it is the *intimacy* of contact which often determines the hazard associated with such potentially reactive mixtures. Decrease in particle size, with a corresponding increase in specific surface, leads to a greater intimacy of contact of components, as well to a greater probability of reaction and to an increase of its velocity. Such an increase of reactivity might cause a LE(low explosive) like BkPdr to explode instead of deflagrating and there is a potential danger in such action. Particularly dangerous are mixtures of chlorates & chlorites with organic matter, sulfur, and/or phosphorus. Also very dangerous are mixtures of perchlorates with reactive metals, such as Mg & Al. They are usually sensitive to impact and to friction and often quite unstable chemically

All other factors remaining constant, those reactions producing the greater volumes of gases are the more dangerous, but on the other hand, it is still necessary to consider the reactivity of the systems

Accdg to Lothrop & Handrick (Ref 2) certain groupings when present in a molecule can either confer upon it or enhance its expl properties. They refer to radicals which confer expl character directly as *plosophors* and those which enhance expl power as *auxoplosive* groupings. The plosophors may lead either to powerful expls or to more sensitive materials and include compds contg the following groupings: $-ONO_2$, $-NH.NO_2$, $=N.NO_2$, $-NO_2$, $-NO$, $-N=N-$, $-O-O-$, and $-N_3$. Organic salts of perchloric, picric, chloric, nitric, bromic, and iodic acids would also be dangerous because of their high oxygen content. Auxoplosive groups include nitrile, oxime, and other linkages

Among the compds which have been neglected as hazardous are nitrations by means of liquid nitrogen tetroxide (N_2O_4) of hydrocarbons, and of its mixtures with various organic substances. For example, a mixture of N_2O_4 70 & NBz 30% had, accdg to A. Stettbacher, a deton vel above 8000m/sec, despite its low density

In the same connection it is pointed out the inherent danger in the use of fuming nitric acid for laboratory nitrations. Aniline and such acid are used in some JATO units

The action of alkalies on nitroparaffins may result in formation of intermediates, leading eventually to salts of fulminic acid

There is also danger in handling of strong hydrogen peroxide, of its salts, and

of their mixtures with organic materials, such as glycerin

On pp 608 & 609 of Ref 3 are listed various dangerous mixtures of inorganic materials, while on p 609 are given 14 precautionary "how to avoid accidents"
Refs: 1) W.R. Tomlinson Jr, K.G. Ottoson & L.F. Audrieth, JACS **71**, 375(1949) 2) W.C. Lothrop & G.R. Handrick, ChemRevs **44**, 419(1949) 3) W.R. Tomlinson Jr & L.F. Audrieth, JChemEduc **27**, 606–09(1950) & CA **45**, 2275(1951) 4) P. Rochlin, "Safety Handbook", Part III. Hazardous Compounds, Mixtures, and Reactions, Expl Res Sect, Expls & Proplnts Lab, Picatinny Arsenal, Dover, NJ (Dec 1959), pp 33–46 (List of compds, mixts, and reactions which are, or may be, dangerous) 5) "Explosive Accident/Incident Abstracts" (A compilation listing description, causes & preventive measures of 219 expl incidents reported to the Armed Services Explosives Safety Board by companies, governmental agencies & other groups from Sept 1961 thru June 1967. The Abstracts are available as ASTIA Document 660020 from the National Technical Information Service, US Dept of Commerce, PO Box 1553, Ravensworth, Va 22151)
NOTE: See also Explosions, Unexpected in this Vol

Explosions, Unique. An expln was caused in Zürich in 1919 during manuf of acetoacetic ester from Ac_2O and EtOH by the violent catalytic activity of $NaHSO_4$ accidentally left in the reaction vessel. Other examples include explosions in the production of endothermic compds (phenyl azide and azodicarbonic ester). Explns from nitro compds particularly in 1920 during distillation of o- and p-nitrotoluenes, explns in the presence of oxygen under high pressure, and explns in the autoxidation of α,α-diphenylethylene, also violent expln following treatment of oxalyl chloride or bromide with Na-K alloy (Ref 1). Explosion catastrophe at Bodio (Tessin) was assumed caused by the spontaneous decompn of addition products of N_2O_4 and unsatd benzenes, so-called nitrosates (Ref 2)
Refs: 1) H. Staudinger, ZAngewChem **35**, 657–9(1922) & CA **17**, 1146(1923) 2) A. Schaarschmidt, ZAngewChem **38**, 537–41 (1925) & CA **19**, 2879(1925)

Explosion Unit Operation (See also "Explosive Disintegration"). The explosion technique has been applied successfully for the modification of substances and for separation of components, as well as for size reduction of materials. This method has been used in rubber reclaiming, wood pulping, manufacture of fiber board, pulverization of minerals and coal and finally for the prepn of puffed cereals

As an example of modification of substances may be cited the devulcanization of rubber scrap (patented by Gross, Ref 2, and the further development by H.F. Palmer). The method consisted essentially in subjecting rubber scrap to high pressure steam for a period of time sufficient to soften and impregnate the mass, and then the pressure was abruptly released. The resulting product consisted of plastic, devulcanized particles, directly suitable for further milling and compounding

As an example of separation of components, the method patented as far back as 1911 by Anderson (Ref 1), may be mentioned. The process involved heating air-dry Indian corn to about 187° in a pressure-tight vessel, then suddenly releasing the pressure. The corn was discharged from the vessel and the hulls and germs were blown off by internal pressure
Refs: 1) A.P. Anderson, USP 990093(1911) 2) R.R. Gross, USP 1963943(1934) (Devulcanization of rubber by explosion) 3) R. Bainer & C.E. Barbee, Agricultural Engineering, Jan 1939 (An internal combustion nut cracker) 4) D. Meigs, ChemMetEngrg **38**, 122–25(Feb 1941) (Explosion unit operation of the process industries) 5) H.L. Ammann, USP 2658821(1953) & CA **48**, 1658(1954) (Apparatus for producing carbon black by the expln process) 6) D. Camici, Metallurgia Ital **48**, 160–70(1956) & CA **50**, 11646(1956) (Slag removal in coke ovens with expls)

Explosions, Unsteady. See Vol 4, p D575 under Detonation, Steady and Nonsteady State

Explosion and Its Utilization. Title of a book which presents a concise & popular exposition of the physical fundamentals of the theory of expln & its action. Great attention is paid to the latest accomplishments in the matter of explns. The author dwells in particular on one of the main problems of modern theory of expln-directed action & control of explns. The simplest designed formulas & numerical data will not only help study deeper the effect of expln, but can also be used in practical application of the acquired knowledge
Ref: G.I. Pokrovskii, "The Explosion and Its Utilization", OTS 60-41, 692, JPRS **5946**, Oct 1960 (English translation of a book entitled "Vzryv i yego Primeneniye", Moscow, 1960, 66pp)

Explosions in Vacuo were discussed by C.K. Thornhill, in ARDE Report **B/30/58**, Nov 1958

Explosion, Velocity of. See Vol 4, pp D629-R to D640-R

Explosion Velocity in Gases. See Vol 4, pp D663-L to D666-L

EXPLOSION (AND DETONATION) WAVES. See Vol 4, pp D676-L to D682-L

Explosion, Work Capacity in. See Vol 4, p D730

EXPLOSIVE. Its definitions by various scientists: M.A. Cook, C.G. Dunkle, K.K. Andreev, F.A. Baum et al and others are given in Vol 4 of Encycl, under "DETONATION, EXPLOSION AND EXPLOSIVES", pp D217-L to D223-R

Explosive 808. A British demolition explosive made in paper-wrapped, 4 oz cartridges 3x1-3/8 inches. The explosive used was "Desensitized Polar Blasting Gelatine", which could be initiated by a primer

Expl 808 could be used for either external or internal work
Note: Compn of "Desensitized Polar Blasting Gelatine" was not found
Ref: Anon, "Explosives and Demolitions", War Department Technical Manual, **FM 5-25**, Washington, DC (1945), p 126

Explosive 851 and Explosive 852. Plastic expls manufd by the Nobel Co in GtBritain

"Explosive Accident/Incident Abstracts". It is the title for a compilation of 219 explosive incidents (each listing description, cause & preventive measures), 110 operational incidents and 38 potential incidents involving expls and other dangerous materials or operations. All reported to the ASESB (Armed Services Explosives Safety Board, Dept of Defense, Washington, DC, 20315)from September 1961 thru June 1967 by companies, government agencies and trade groups. Sample titles: "Explosion During Mixing Experimental Propellant"; "Explosion During Casting and During Operation"; "Inert Gas Leak"; "Inhalation of Hydrogen Cyanide Fumes"; "Explosion of Pyrotechnic Devices", etc. Each organization reporting its incidents to ASESB receives from it anonymous reports from all other cooperating organizations. These reports are valuable because they alert operators to hazards of which they might not have been aware. The Franklin Institute Research Laboratories have joined ASESB's group of cooperators, and the newsletters of "Explosives and Pyrotechnics" will excerpt future accident/incident reports for the benefit of its readers

The 6-year compilation is available as **AD 660 020** from National Technical Information Service, PO Box 1553, Ravensworth, Va 22151
Ref: Gunther Cohn, Edit, Expls & Pyrots 1 (6), 1968

Explosive Actuator XE-16A was developed at NAVORD Labs to replace the Mk 1 Mod 0 actuator for use in Mk 52 drill mine. Better surveillance characteristics were obtd by using Unique Powder (a smokeless proplnt of unspecified compn) as a substitute for Black Powder formerly used as the base chge. Milled normal Pb Styphnate was substituted for DDNP-Potassium Chlorate mix as the ignition chge. Several design changes to increase ruggedness were also made. Laboratory & field tests indicated that the XE-16A Actuator would be a satisfactory actuator in the Mk 52 Drill Mine
Ref: E.E. Kilmer & M.J. Falbo, NAVORD Rept **6111**(1958)

Explosive Actuator, Re-chargeable. An experimental linear actuator similar to the "explosive diode" (qv) places a steel bulkhead between its donor charge (fired via detonating cord) and receptor charge (which fires an explosive, pyrotechnic or propellant). Bulkhead transmits the donor's shock wave apparently with no damage, so actuator can be re-charged and used repeatedly. Developed by North American Aviation for NASA-Houston, Product Engineering, April 8, 1968
Ref: G. Cohn, Edit, Expls&Pyrots **1**(5), 1968

Explosive Area. Any area in which expls, propellants, pyrotechnics or ammunition are manufd, stored, handled, shipped, maintained, renovated, salvaged, etc is called "explosive area". This includes plant buildings and magazines. The area should be surrounded by a fence with guards stationed around it and the gates to prevent any unauthorized persons from entering the area. Within the area, precautions should be taken to guard against fire and accidents
Refs: 1) Anon, "Ammunition Inspection Guide", **TM 9-1904**(1944) 2) Anon, "Safety Manual", **AMCR 385-100**(April 1970), p 2-4

Explosive Assembly and Method of Making It. The device described in USP 2541334(1951) of C.H. Carey & D.R. Walton is intended primarily for expls contg a high proportion of AN and little or no NG. In expls of this kind, as the apparent density is increased (as by hard packing), there is reduction in sensitivity to detonation and decrease in sensitivity by influence. Introduction of an auxiliary expl (booster) compressed to a lesser degree than the main charge assures complete detonation. It also permits the use of insensitive expls (as main charges) which could not be detonated by the cap alone

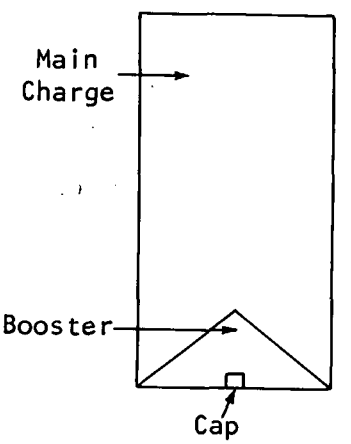


FIG Ex2

The power producing expl assembly consists of a rigid cylindrical container (Fig Ex2); a rigid conical partition having an open base spanning said container is freely resting against one end wall of the container. This cone is extending toward the opposite end of container and has an apex angle between 60° and 120°. A booster expl fills the space below conical partition, while space above it is filled with main charge. A receptacle for a detonator element is adjacent to booster

A rather complicated method for prepg such an assembly is described in the patent, but is not given here

Explosive of Audrieth. Mixture of AN 78.5–83.5, hydrazine nitrate ($N_2H_4.HNO_3$) 5–10, TNT 7.5, Al powder 0.5, pulverized carbon 3 & chalk 0.5% ‹ It is claimed that hydrazine nitrate increased considerably the sensitivity and the rate of detonation of the mixt
Refs: 1) L.F. Audrieth, USP 2704706(1955) 2) Giua, Trattato **6**(1)(1959), 374-L

Explosive B & T No 21 (Victorite). A compn prepd by mixing flour (18.8), sulfur (1.9) & $KClO_3$ (75.1%) in a soln of resin (4.2%) in an org solv, and then removing the solv by evaporation. This process is designed to coat the expl particles with resin, thereby waterproofing them and reducing their sensitivity. The product may also be dyed different colors. The compn was unsatisfactory for military application because of its sensitivity, hygroscopicity & low brisance
Ref: J.D. Hopper, PATR **481**(1934)

Explosive Bolts. See under Explosive Rivets

Explosive Bonding, also called **Explosive Cladding.** See Detaclad Process in Vol **3**, pp D96-L to D97

Explosive Bonding Analog is described by J.E. Kowalick & D.R. Hay in Frankford Arsenal, Report **A71–7**, Aug 1971, entitled "A Flow Analog for Explosive Bonding". Order as AD 730299 from NTIS, US Dept of Commerce, PO Box 1553, Ravensworth, Va 22151. Abstracted in Expls&Pyrots **5**(3) 1972

"Explosive Bonding Gets Biggest Job Yet". Title of brief paper in C&EN **42**, Aug 31, 1964, p 46. It describes achievements of DuPont in clad-plate market and the fabrication by Nooter Corp of St Louis, Mo of four chemical process vessels for Amoco, using DuPont's titanium-clad plates

Explosive Bonding, Mechanism of. An aerohydrodynamic analogy is used to construct a description of the formation of wavy interfaces during expl bonding, which is described by J.T. Kowalick & D.R. Hay in Metallurgical Transactions, Vol **2**, July 1971, pp 1953–58
Ref: G. Cohn Edit, Expls&Pyrots **4**(11), 1971 (Abstract)

Explosive Bonding, Nature of. Wave formation in expl bonding is viewed as a special case of the transition from laminar to a turbulent metal flow regime set up along the collision region between two metal interfaces. Employing a novel semicircular technique, dynamic parameters of the process are used to determine Reynolds numbers for dynamic metal flow. They are correlated with conditions for obtaining wavy bonded interfaces between metal, the most desirable type of bonding from the standpoint of obtg superior mechanical properties. A Reynolds number range of 0.38 to 14.7 is established as a necessary and sufficient criterion for expl bonding
Refs: 1) J.F. Kowalick & D.R. Hay, "The Nature and Formation of the Bond in the Explosive Bonding of Metals", Frankford Arsenal, Report **R–1966**, June 1970. Order as AD 714221 from NTIS, US Dept of Commerce, PO Box 1553, Ravensworth, Va 22151 2) Gunther Cohn, Edit, Expls&Pyrots 4(6), 1971 (Abstract)

"Explosive Bonding Technique Emerging in NASA Plans". Title of the paper by Bob Ward in Metalworking News, Oct 13, 1969, p 27. Progress is reported in expl bonding of filament reinforced metallic composites for possible use in rocket and space station structures. Focus has been on lightweight composites reinforced with stainless and low carbon steel wire. NGu (Nitroguanidine) served as the driving expl in forming rods from expl compaction of a matrix of high-strength wires surrounded with powdered metals. Plates of widely varying metals

were bonded with similar procedures. Tensile strength of up to 70000 psi has been obtd with 1100-0 Al properly reinforced
Ref: Gunther Cohn, Edit, Expls&Pyrots **3** (1), 1970 (Abstract)

Explosive Branding Iron. A device for permanently marking animals, birds, fishes, etc required in wildlife studies on specific mortality, and fecundity rates. Total device weighs 3 pounds and the expl charges for 200 single-digit brands weigh 5 pounds. The applicator consists of a modified spear-fishing gun fitted with a pivoted rubber template which holds the brand. Branding is accomplished by setting off a piece of MDF (Mild Detonating Fuse) shaped to fit the template cavity which is a mirror image of the brand. MDF is 2 grains PETN per foot. Initiation is by a commercial percussion detonator fired by the spear-fishing gun trigger. The brand has been successfully applied to seals with no animal showing any distress

Most of the previously available branding devices have the disadvantage of being either extremely heavy and cumbersome, or in the case of freeze-branding, the impossibility of being kept supplied in the field with the necessary cryogen. Electric branding needs a cumbersome high-voltage generator
Refs: 1) R.E. Homestead et al, "A Portable, Instantaneous Branding Device for Permanent Identification of Wildlife", JourWildlife-Management **36**(3), 1972, pp 947–49 2) G. Cohn, Edit, Expls&Pyrots **6**(6), 1973

Explosive BTM of Fleischnick. See BTM in Vol **2** of Encycl, p B319-R

Explosive of Buccianelli consists of a white precipitate obtd on adding triethanolamine soln to a soln of Pb perchlorate
Ref: E. Buccianelli, ItalP 588789 (1959) & CA **54**, 6130 (1960)

Explosive Bullets or Shell Bullets. A brief description, including historical background is given in Vol **2**, p B326, under BULLETS. There is also a brief description of "Bullets, Dumdum" on p B331-R

A more complete description of Explosive Bullets is given in this Vol under "Explosive Cartridges" and under "Explosive and Explosive Incendiary Ammunition"

Explosive C–4 or Composition C–4, also known as **Harrisite,** was developed before 1950 at Picatinny Arsenal by K.G. Ottoson. Its prepn and props are described in Vol **3** of Encycl, p C485

C–4 was employed during the Vietnam war for blowing up captured enemy bunkers and to clear trees for helicopter landing areas, and then it was discovered by some soldiers that eating of C–4 produced a better "high" than marijuana. Unfortunately, the after effects were very bad: convulsions and kidney infections, with brain damage that could be permanent. Accdg to Lt Col A.J. Ognibane of the Army Medical Corps, the hospitals were full of soldiers who had eaten the stuff. The situation became so bad that many infantry companies were not being issued C–4 any more. The Captain had to have a special demolition expert to bring C–4 in by chopper when they needed it. Some outfits had difficulty getting needed supplies because the choppers could not land
Refs: 1) Anon, American Pyrotechnic Fireworks News, Vol **4**, No 2, Feb 1971 2) G. Cohn, Expls&Pyrots **4**(7), 1971

Explosive, Canadian. An aqueous slurry of AN and TNT, developed by Canadian Industries Ltd in collaboration with inventors M.A. Cook and H.E. Farnam has been used for open pit blasting at Iron Ore Co of Canada's Knob Lake operations in Newfoundland. The fluid character of the material enables it to be loaded efficiently into bags or directly into the borehole without a container. Because of its high density (1.4) and good water resistance, it can be loaded

under water and performs effectively under wet conditions. Its strength is 70% of Gelatin Dynamite but it is considerably less sensitive to initiation. It requires a 6-oz booster of Pentolite with an electric blasting cap or Primacord detonating fuse. The explosive's high strength and efficient loading characteristics are expected to make it especially suited to hard breaking conditions in open pit mines
Ref: Anon, C&EN, Nov 17, 1958, p 43

Explosive Capsule. An expl charge is made by loading the initiating expl into a small cap, which it partially fills, and then pressing the main expl charge onto this in the same mold. The cap, which then contains all of the initiating expl and part of the main charge, serves to hold the two together
Ref: Poudreries réunis de Belgique SA, BelgP 528984 (1954) & CA **52**, 5825 (1958)

Explosive Cartridge. A definition found in our notes says that such a cartridge contains a regular expl charge and, in a separate casing, an admixture of substances having high burning temperatures, such as Al, Mg, ferrosilicon or red P. No example of such cartridges is known to us. On the other hand, Yust, in the paper published in "American Rifleman", (See Ref), described under the title "Rifle Caliber Explosive Cartridges" the items which should be called "Rifle Cartridges with Explosive Bullets"

The following items are included in Yust's paper:
1) Argentine 7.65mm, Type R, HE cartridge manufd in Austria before WWI by Hirtenberg Patronenfabrik (See Fig 1 of Ref)
2) Austrian cartridges, Model 1910 and Model 1914, known as Einschusspatronen, were manufd at the government Arsenal at Wellersdorf. The expl in the 1910 Model consisted of $KClO_3$ & Sb_2S_3, while in the 1914 Model it was compressed BkPdr mixed with Al (Fig 2 of Ref)
3) Austrian cartridge of 1917, known as "airburst shrapnel" was included for use in machine guns (Fig 3 of Ref)
4) Austrian "antiballoon cartridges" of 1915 & 1916 were used in Mannlicher rifles (Fig 4 of Ref)
5) French "spot-light cartridges" in 8mm Lebel rifle existed in 2 patterns. Their expls placed in the cavity of bullet nose, was sensitive to detonation on impact (Figs 5 & 6 of Ref)
6) German 7.92 x 57mm WWI cartridges were of three types. Jackets of their bullets were of uncoated steel (Fig 7 of Ref)
7) German 7.92 x 57mm incendiary cartridge introduced shortly before WWII was known as B-Patrone. It was used as an observation cartridge for training purposes (Fig 8 of Ref)
8) German 7.92 x 57mm training cartridge which contd an explosive element, was known as "S.m.K. L'spur Ub.m.Z" (Spitzgeschoss mit Stahlkern und Leuchtspur Ubung mit Zerstorer), which stands for "Pointed Bullet with Steelcore and Tracer, Practice, Self-Destroying", translated by Yust as "Practice Ball with Steel Core and Self-Destroying Tracer" (Fig 9 of Ref)
9) Italian 6.5mm expl cartridge of WWI could be recognized by an unusual shape of cupronickel-jacketed bullet (Fig 10 of Ref)
10) Japanese Navy 7.7mm rimmed expl cartridge of WWII was used in Naval machine guns, ground machine guns and was suitable for use in British .303 rifles and machine guns. The bullet had a cupronickel jacket. The front compartment inside the nose contd RDX or PETN; to the rear of this was a gilding-metal cup filled with Al & RDX or with Al & TNT. This was backed by a lead plug (Fig 11 of Ref)
11) Japanese Army 7.7mm, semi-rimmed expl round with flat-nosed, gilding-metal jacketed bullet is shown in Fig 12 of Ref
12) Japanese Army 7.92 x 57mm expl round which had a flat-nosed, gilding metal-jacketed bullet is shown in Fig 13 of Ref
13) Imperial Russian 7.62mm expl round for rifles and machine guns had a cupronickel jacketed bullet with flat nose. It contd the expl, detonator and firing pin (Fig 14 of Ref)
14) Soviet Russian 7.62mm incendiary cartridge had the gilding-metal clad, steel-

jacketed bullet. The front of the bullet contained thermite powder, followed by a lead sheath contg a detonator backed by a firing pin. A lead plug was in the base. Cartridge cases were either brass or copper-coated steel (Fig 15 of Ref)
15) United States .30 Gov't M1906 H.E. Pomeroy expl cartridge, made by Winchester Co for the Government, is shown in Fig 16 of Ref. Its bullet had a pointed Cu tube inserted in the front end, which contd the expl charge
16) United States .30 Gov't M1906 HE Spot-Light round is shown in Fig 17 of Ref
17) United States 11mm Machine Gun HE Spot-Light round is shown in Fig 18 of Ref
(See also under "Explosive Bullets" in this Vol)
Note: Later models of US expl bullets are described in this Vol, under "Explosive and Explosive Incendiary Ammunition"
Ref: Ch.H. Yust, Jr, "Rifle Caliber Explosive Cartridges", American Rifleman, January 1963, pp 22–4

Explosive Catapults are devices used for launching naval shipboard airplanes. Due to the lack of space aboard ships, it is impossible to have long enough runways to properly launch planes by ordinary means. Therefore, a catapult is used to impart sufficient initial velocity to the plane to launch it properly. Formerly, such catapults were operated by compressed air, but now expls are used. One method for launching planes is to fasten on the under side of the wings of a plane some rockets and then fire them at take-off time. This is now known as RATO (rocket-assisted take-off), but was formerly known as JATO (jet-assisted take-off) (Refs 1 & 3)

Catapults ejecting pilots and other personnel from rapidly travelling airplanes are ejected by cartridges contg propellants. They are known as CADs (cartridge-actuated devices) (Ref 2)

The general term for an auxiliary rocket engine specifically used for providing extra thrust to a heavily-loaded aircraft during take-off run and initial climb is ATO (assisted take-off) (Ref 1)
Refs: 1) Encycl PATR **2700**, Vol **1** (1960), p A197-R [Assisted Take-off (Units)]
2) Encycl PATR **2700**, Vol **2** (1962), pp C70-R to C72-R [Cartridges and Cartridge-Actuated Devices (CADs), called Propellant Actuated Devices] 3) Encycl, PATR **2700**, Vol **2** (1962), p C91-R [Catapult (Modern)]

Explosive Characteristics, Calculation of. Roth (Ref) described calculation of technically important expl characteristics by means of thermodynamic and hydrodynamic laws from known values of composition, heats of formation and densities
Ref: J. Roth, SS **35**, 193–96, 220–21, 243–45 (1940) & CA **35**, 1635 (1941)

Explosive Cladding. See Explosive Bonding, Explosive Bonding Analog, Explosive Bonding Gets Bigger Job Yet, Explosive Bonding Mechanism of, Explosive Bonding Nature and Explosive Bonding Technique in this Vol

Also an item, *Explosive Cladding*, is abstracted by G. Cohn Edit, in Expls&Pyrots **2** (2), 1969 from Metalworking News via Technical Survey, Nov 30, 1969:
"Cladding the interiors of vats, containers for storage of corrosive substances, and even sheet metal tubs by explosive bonding techniques is becoming more popular in West Germany. In cases where high temperatures, with or without pressure, are involved, savings in structural costs up to 75% can be obtained. Today, almost all thickness combinations are possible with titanium on steel. These containers are also welded explosively"

Explosive Combustion and Deflagration. See under Detonative (or Explosive) Combustion or Explosive Deflagration in Vol **4** of Encycl, p D731-L & R

Explosive Compaction. See under Explosive Fabrication of Metals

Explosive Compositions are mixtures of explosive and non-explosive substances. They are listed in this Encycl as individual items such as Amatols, Ammonals, Ammonium Nitrate Blasting Explosives, Black Powder, Compositions A, B, C & D, Cyclonites, Dynamites and as Explosive Compositions listed further in this Vol. (See also Composite Propellants described in Vol 3, pp C464 to C473)

Explosive Composition 146-4-G is listed in Vol 3 of Encycl, p C474-L and described by E.T. Benning in PATR 2062(1954)

Explosive Compositions I & II are described in Vol 3, p C474-L and by F.H. Fogel & S. Sage in PATR 1431(1944)

Explosive Compositions A, A-2, A-3 and A-4. See Compositions A, A-2, A-3 and A-4 under Composition A Type Explosives in Vol 3, p C474-R & C475-L. Also Specification MIL-C-440B

Explosive Composition A-5. It is described as *Composition A-5* in Vol 3 of Encycl, pp C476-R to C477-R. At the time of description there was no Specification, but Purchase Description **PA-PD-2321** (1961), entitled Explosive Composition A-5. There existed at that time two types: I and II

In the Specification MIL-E-14970A(MU) 6 Sept 1970, superseding MIL-E-14970(MU), 20 Jan 1969, it is stated that A-5 shall consist of RDX Type A or B, complying with the requirements of Spec MIL-R-398 and Stearic Acid, complying with the requirements of Spec MIL-S-271. The composition shall be made by coating the RDX with stearic acid by the wet slurry method (See Note after procedure 4.4.5. Determination of Bulk Density)

Table Ex3

Composition:

Ingredients	Percent Min	Max
RDX	98.5	99.0
Stearic Acid	1.0	1.5
Moisture	–	0.10
Insoluble Particles	Not more than 5 particles retained on No 60 Sieve and none on No 40	
Bulk Density	Min 0.95g/cc for Type I and 1.0 for Type II	
Granulation	Min 99.0% thru No 12 Sieve and max 2.4% thru No 200 Sieve	

Tests:

4.4.1.1 Determination of Stearic Acid. Use the same procedure as item d on p D477-L of Vol 3. Add at the end: Repeat the above extraction, drying and weighing procedure with individual 20ml portions of chloroform until total loss in weight on subsequent extractions is less than 0.003g. Calculate the loss in weight of the crucible plus contents to % stearic acid on a moisture-free basis using the formula given on p D474-L of Encycl

4.4.1.1.1. Determination of Stearic Acid by Alternate Method which is described in Amendment 1, 24 Jan 1973 to MIL-E-14970A(MU)

Add approx 5g of the prepd sample into a previously tared clean, dry, medium porosity crucible, contg a filter pad. Reweigh crucible and contents on an analytical balance and record the total wt to the nearest 0.0002g. Place the crucible contg sample into a stainless steel pan located under a fume hood, and add 20ml of aliphatic naphtha to the crucible. Allow the solvent to remain in contact with sample at RT for about 5 minutes, while stirring the slurry and breaking-up any lumps. Remove the crucible from the pan and place it in the rubber ring located in the neck of a vacuum suction flask placed under

the hood. Suction filter the solvent and then repeat twice the above operations of addg 20ml naphtha, extracting for 5 minutes and filtering. Rinse the interior of the crucible with approx 20ml of naphtha from a wash bottle and aspirate the residue until the odor of naphtha is no longer detectable (approx 2 mins). Dry the crucible with residue in an oven at 90° to 105° for approx 30 mins, remove from oven, cool in a desiccator at RT and weigh to the nearest 0.0002g. Calculate % of stearic acid on a moisture free basis as follows:

$$\% \text{ Stearic Acid} = \frac{10(X-Y)}{W}$$

where: X = Initial weight of crucible with sample
Y = Final wt, after extraction and drying
W = Weight of sample on a moisture free basis

4.4.1.2 Determination of RDX.
Subtract percent of Stearic Acid from 100 percent

4.4.2 Determination of Insoluble Matter.
Use Method 106.1 of MIL-STD-650 with acetone as solvent: Weigh ca 50g of the expl to within 10mg, place in a 400-ml beaker and add 100ml of acetone. Heat on a steam bath until all lumps are broken and all soluble material is dissolved. Pour the mixture thru a small US Standard Sieve No 60 and wash the insol matter from the beaker onto the sieve with a stream of acetone. Wash the residue on the sieve with addnl acetone to remove the undissolved soluble material. Dry the sieve and count and examine any particles remaining on the sieve. Brush any particles retained on No 60 sieve onto No 40 sieve. Note if the particles are grit as indicated by their lack of uniformity and a scratching noise when pressing and rubbing the material on a smooth glass slide with a smooth steel spatula. Report the number of particles on No 60 sieve and on No 40 sieve

4.4.3.1 Determination of Moisture
Use Karl Fischer Method, described in MIL-STD-650 as Method 101.4, with methanol as the special solvent. A detailed description of the Karl Fischer Method is given under DYNAMITE in Vol 5 of Encycl, pp D1623 to D1627

4.4.3.2 Determination of Moisture by Alternate Method. This includes moisture and volatiles. For rapid determination use the "oven method" described in MIL-STD-650 as Method 101.5:
Spread a 5g sample, weighed to within 0.2mg, in a tared weighing dish (Al or glass), diam 60mm, depth 30mm, provided with a stopper. Heat in an oven at 100–105° for 1 hour, cover the dish and cool it to RT in a desiccator, contg an indicating desiccant. Reweigh the dish with sample and determine the loss of wt, A, subtracting dish + dry sample from dish + moist sample

% Moisture = 100A/W, where W = weight of moist sample

Note: In case of non-agreement, the Karl Fischer Method shall be the standard

4.4.4. Determination of Granulation
Place a sample of 50±0.01g on a US Standard Sieve No 12 which is superimposed on top of a Sieve No 200, which is assembled with a bottom pan. Cover the upper sieve and shake the ensemble for 3 minutes by means of a mechanically operated (by remote control with adequate shielding) sieve shaker, which shall impart to the sieves a rotary, uniform motion and tapping action of 300±15 gyrations and 150±10 taps of the striker per minute. Weigh the portions (in grams) retained on or passed by the sieves and calculate the results to a percentage basis as described in Method 204.1 in MIL-STD-650:

% Retained = (A + B) 100/W
% Through = [W − (A + B)] 100/W

where: A = Weight retained on designated sieve
B = Weight retained on sieves nested above designated sieve
W = Weight of sample

4.4.5. Determination of Bulk Density
Use the apparatus consisting of a plywood box, 4x4x4 inches inside dimensions; padded inside top and bottom with ¼-inch rubber foam of approx 0.029g/cc bulk density and provided in hinged top with a hole to fit a 250-ml graduated cylinder

Procedure: Weigh 200g sample and pour it into 250-ml graduated cylinder. Insert the cylinder thru the above mentioned hole and close the box. Lift the cylinder until its bottom flange contacts the top of the box. Then drop the cylinder, again lift and drop it until 20 drops have been made, at the rate of one every 5 seconds. Read the volume of compacted powder in the cylinder

Calculate: Bulk Density = A/B

where: A = Weight of sample in grams

B = Volume of compacted sample in ml

Note: The following *wet slurry method of coating* has been found satisfactory. Add molten stearic acid at approx 70°C to a hot water slurry of RDX at approx 70° and then, after thorough mixing, cool, filter and dry the coated product

Explosive Composition APX. See APX Explosive in Vol **1**, p A475-L

Explosive Compositions B, B-2, B-3, B-4, B Desensitized and Cyclotols. See under Composition B Type Explosives and Cyclotols in Vol **3** of Encycl, pp C477-R to C482-L. Also Specification MIL-C-401D, MIL-C-45113A-(MU) and MIL-C-46652(MU)

Explosive Compositions, Brisant (of High Density). The following expl compns contg solid TNT and liquid nitric esters are described. Nitroglycerin 15–20, soluble Nitrocellulose 0–7.5, Kieselguhr 0–5, stabilizer 0–2.5, TNT 85–70%. The TNT is in the form of crushed flakes

Ref: J. Taylor and S.H. Davidson, BritP 578371 (1946) & CA **41**, 1842 (1947)

Explosive Compositions C, C-2, C-3 and C-4 (Harrisite). See under Compositions C Type Explosives in Vol **3**, p C484-L to C488-L. Specification for C-3 is MIL-C-427A and for C-4 MIL-C-45010A(MU)

Explosive Compositions Cyclotols. See Tables on pp C479 & C480 in Vol **3** under Composition B Type Explosives and Cyclotols. Also Specification MIL-C-13477B(MU)

Explosive Composition D-2. See Composition D-2 in Vol **3**, p C488-R

Explosive Compositions EL-387A and EL-387B of DuPont Co. See Compositions EL-387A and EL-387B in Vol **3**, pp C488-R to C489-R

Explosive Composition of Gordon and Little. Heterogeneous expl compns are made by mixing NH_4NO_3 with an org fuel capable of forming a homogeneous melt below 170°, such as urea, hexamethylene tetramine or dicyandiamide, to form an O-balanced mixt. Mixt is quickly melted and then quickly cooled so that the crystals of ammonium nitrate have dimensions of $<100\mu$ and are embedded in a matrix of fuel

Ref: W.E. Gordon & A.D. Little, BritP 1014071 (1965) & CA **64**, 7962 (1966)

Explosive Composition H-6. See H-6 Explosive in Vol **7** and in AMCP **706–177** (1971), pp 146–49. Also Specification MIL-R-21723

Explosive Compositions HBX-1 and HBX-2. See HBX-1 and HBX-3 Explosives in Vol **7** and in AMCP **706-177** (1971), pp 156 to 163

Explosive Compositions, HBX Type. See HBX Type Explosives in Vol **7** and Specification MIL-E-22267A, 31 May, 1963

Explosive Compositions HEX-24 and HEX-48. See HEX-24 and HEX-48 Explosives in Vol **7** and in AMCP **706–177** (1967), pp 164 to 169

Explosive Composition of Hradel. Solid expl mixts are produced by mixing 70-3% by wt NH_4NO_3 in soln with NH_3, H_2O, or their mixts with 27-30% of a light metal, such as Al or Mg. Components react exothermally during 0.5-9 hrs to form a solid expl . Preferably the metal is in a particular form. Detonation can be done by means of a shaped charge
Ref: J.R. Hradel, USP 3177102 (1965) & CA **63**, 426 (1965)

Explosive Composition HTA-3. See HTA-3 Explosive in Vol **7** and specification MIL-E-46495A (MU), 30 Nov 1964 with Amendment 1, 5 April 1972. Also in AMCP 706-177 (1967), pp 178 to 181

Explosive Composition Minok-2 (and Minols 1 & 3). See Minol-2 in AMCP **706-177** (1971), pp 209 to 212, where also are given compns of Minols 1 & 3 but no properties. Specification for Minol-2 is MIL-M-14745 (MU)

Explosive Compositions of Monsanto Co. A sensitive and powerful blasting expl is obtained by mixing 6 parts of a light fuel oil with 94 parts hi gh d microprills having a bulk d of $\leqslant$100lbs/cu ft and contg $\leqslant$94% NH_4NO_3, $\leqslant$ 5% of a hydratable salt, such as $Mg(NO_3)_3$, $AL(NO_3)_3$, $Ca(NO_3)_2$ and $MgSO_4$ to impart dimensional stability during storage; and $\leqslant$5% H_2O
Ref: Monsanto Co, BritP 988095 (1965) & CA **63**, 1650 (1965)

Explosive Compositions MOX. See MOX-1, 2B, 3B, 4B and 6B in AMCP **706-177** (1971), pp 213 to 225

Explosive Compositions from Nitrated Toluene and Naphthalene. A soln of 30% $C_{10}H_8$ and 70% boluene is nitrated with 2.5 times its wt of an equal mixt of sulfuric and nitric acids at 130° to give a product (I) mp 60° and contg 16.9%N. An expl is made by mixing $C_3H_5(ONO_2)_3$ 20, Nitrocellulose 0.8, wood powder 2, NH_4NO_3 71.2 and I 6.0%
Ref: K. Yamasue et al, JapanP 2097 (1954) & CA **49**, 2076 (1955)

Explosive Composition NSX (Nitrostarch Demolition Explosive). See AMCP **706-177** (1971), pp 246 to 248

Explosive Compositions Octols. See Octol 70/30 and Octol 75/25 in AMCP **706-177** (1971), pp 247 to 258. Also Spec MIL-O-45445A

Explosive Compositions for Oil Well Gun Perforators. Expl charges consisting of the Pb Salt (I) of ethylenedinitramine or in combination with other heat stable propellants are suitable for deep well operations. This compd does not spontaneously explode under about 300° and it is a relatively powerful expl by itself. Low impact and friction sensitivity, moderate power or brisance, produces large vols of gas, high flash point
Ref: A.O. Franz, USP 2708623 (1955) & CA **49**, 14326 (1955)

Explosive Composition PB-RDX. See PB-RDX in AMCP **706-177** (1971), pp 259 to 264

Explosive Composition PBXN-5. It consists of HMX 95 & Viton (vinylidene fluoride hexafluoropropylene copolymer) 5%, and has a rate of deton of 8760m/sec at density 1.82g/cc. Its military requirements are given in Specification MIL-E-81111A, "Explosive, Plastic-Bonded Molding Powder (Feb 1973)

Explosive Compositions Pentolites. See Pentolites 50/50 and 10/90 in AMCP **706-177** (1971), pp 272 to 275

Explosive Composition Picratol 52/48. See Picratol 52/48 in AMCP **706-177** (1971), pp 284 to 287

Explosive Composition PIPE. See PIPE in AMCP **706-177** (1971), pp 294 to 295

Explosive Composition Plumbatol. See Plumbatol in AMCP **706-177** (1971), pp 296 to 297

Explosive Composition PLX (Liquid). See PLX (Liquid) in AMCP **706-177** (1971), pp 298-301

Explosive Compositions PTX-1 and PTX-2. See PTX-1 and PTX-2 in AMCP **706-177** (1971), pp 306 to 311

Explosive Composition PVA-4. See PVA-4 in AMCP **706-177** (1971), pp 312 to 314

Explosive Composition of Pyrochemie. It consists of 60-95 parts by wt of "explosive acid" and 5-40 of a gelation agent. The acid is obtd by mixing 50-85 parts by wt of nitric acid, preferably d 1.52 (contg $ONOSO_3H$ and having N_2O_4 content 10 to 35%) and 15-50ps concd sulfuric acid contg oxidizable org or inorg substances (such as DNT, TNT, SiC, Ba or Amm sulfates). The gelation agent consists of finely divided voluminous SiO_2 having an apparent d preferably 0.040kg/cu dm or of silicates of Al, Mg, Ca, Ba or Sr. The ratio $ONOSO_3H:H_2SO_4$ in acid is preferably >1.5:1
Ref: "Pyrochemie" Studien- und Patentverwertungs-G.m.b.H., AustrianP 193775 (1957) & CA **52**, 2410 (1958)

Explosive Composition of Rinkenbach & Carroll: AN 79-84 & dry urea 21-16%; deton velocity 2900m/sec; equivalent in strength to 50% Dynamite
Refs: 1) W.H. Rinkenbach & W.J. Carroll Jr, USP 2814555 (1957) 2) Giua, Trattato **6**(1), 375 (1959)

Explosive Composition RIPE. See RIPE in AMCP **706-177** (1971), pp 318 to 319

Explosive Composition of Sato. The expl consists of nitrourea 35-60, AN 20-40, NG 3-20 and urea 1-5%
Ref: J. Sato, JapanP 296 (1954) & CA **48**, 13222 (1954)

Explosive Composition of Scott. Detonatable expl compns contg uncoated, prilled ammonium nitrate and uncoated, prilled calcium nitrate are discussed. Mixts contd 1-70% by wt of calcium nitrate and 4.5-8.46 % by wt of carbonaceous fuel such as diesel oil. Combinations contg 60 wt% NH_4NO_3 with both 10-30 wt% of $Ca(NO_3)_2$ and 10-30 wt% $NaNO_3$ are also described
Ref: E.M. Scott Jr, USP 3180768 (1965) & CA **63**, 426 (1965)

Explosive Composition T-9. See Composition T-9 in Vol 3 of Encycl, p C490-L

Explosive Composition of Tetrytols. See Tetrytols 80/20, 75/25, 70/30 and 65/35 in AMCP **706-177** (1971), pp 341 to 349

Explosive Composition of Torpex. See Torpex in AMCP **706-177** (1971), pp 359 to 363

Explosive Composition Trimonite. See Trimonite in AMCP **706-177** (1971), pp 370 to 372

Explosive Composition Tritonal 80/20. See Tritonal 80/20 in AMCP **706-177** (1971), p 386 to 390

Explosive Composition Veltex No 448. See Veltex No 448 in AMCP **706-177** (1971), p 391 to 394

EXPLOSIVE D, AMMONIUM PICRATE OR DUNNITE. See 2,4,6-TRINITROPHENOL, AMMONIUM SALT under PHENOL AND DERIVATIVES

Explosive Conditioning. Mentioned, but not described, under Explosive Fabrication of Metals

Explosive Decomposition, Rate of. Using a special apparatus, which is a modification of that used by the US Ordnance Dept (See Fig Ex3), Henkin & McGill (Ref) determined expln temperature at various times of exposure. For expls which ignite or deflagrate on heating, 25mg samples were used and the results for several expls are listed in Table Ex 4

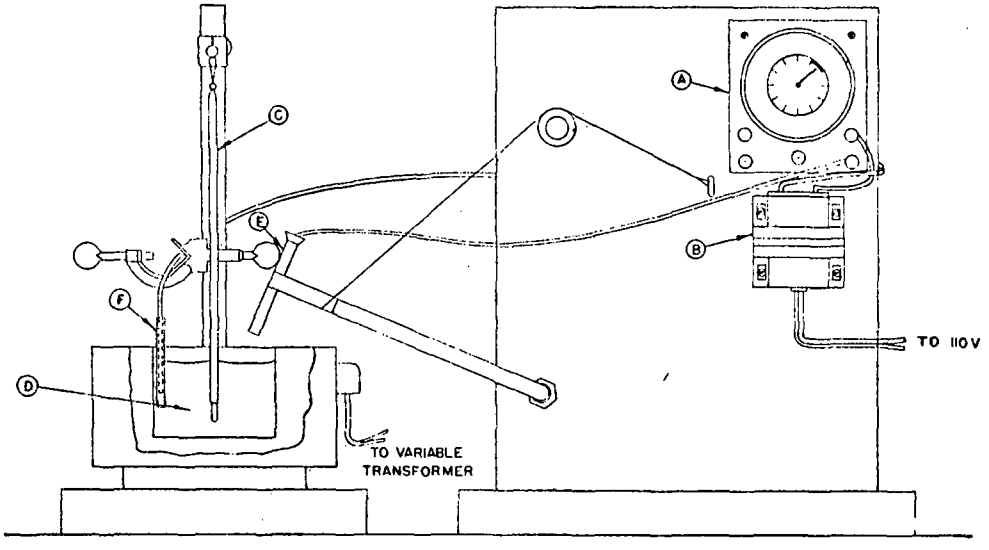


FIG Ex3. DIAGRAM OF APPARATUS

Table Ex4

Substance	Explosion Temp, °C	Exposure Time, Seconds
Tetryl	346	0.325
(Trinitrophenyl-	314	0.742
methylnitramine)	285	1.45
	269	2.22
	264	No action
EDNA or	314	0.166
Haleite	251	0.45
(Ethylenedi-	203	1.18
nitramine)	180	4.88
	162	37.1
	158	120.
	149	793.
	142	No action
BkPdr	359	12.0
(Black Powder)	350	15.4
	320	34.1
	315	No action
NC (13.4%N)	292	0.38
(Nitrocellulose)	264	1.30
	234	5.80
	198	41.4
	174	120.
	170	No action
NC(12.6%N)	350	1.48
	312	5.50
	286	16.6
	267	50.3
	260	No action
PA Picric Acid)	350	1.48
	315	5.50
	277	26.3
	267	50.3
	260	No action

For expls which detonate on heating, 3mg samples were used and results are shown in Table Ex5

Table Ex5

Substance	Explosion Temp, °C	Exposure Time, Seconds
MF	304	0.108
(Mercuric	238	1.03
Fulminate)	200	7.55
	176	105.
	170	No action
LA	360	0.560
(Lead Azide)	341	0.865
	319	1.55
	314	No action
DADNP	247	0.133
(Diazodinitro-	200	0.725
phenol)	177	5.00
	168	16.
	160	No action
LSt	328	0.56
(Lead Styphnate)	301	4.47
	280	41.3
	275	No action
NG	261	0.108
(Nitroglycerin)	230	0.675
	211	3.75
	205	No action
ErTeN	275	0.108
(Erythritol	239	0.475
Tetranitrate)	205	4.95
	200	No action

From the values of Tables Ex4 and Ex5 were estimated lowest expln temps, expln temps at exposure time of one second and the activation energy E in kcal, using the formula:

$\log t = E/RT + \text{constant}$

Results are listed in Table Ex6

Table Ex6

Substance	Expln Temp, °C Lowest	After 1 sec	Energy, E for Lowest Temp	Highest Temp
Tetryl	265	300	–	27.4
EDNA	150	205	80	10.0
BkPdr	320	>360	–	20.6
NC(12.6%N)	175	270	67.0	26.5
PA	265	360	–	27.4
MF	175	240	58.0	20.2
LA	315	340	–	21.1
DADNP	165	195	48.0	29.0
LSt	280	320	–	58.8
NG	210	220	–	22.6
ErTeN	205	220	51.1	22.8

Ref: H. Henkin & R. McGill, IEC **44**, 1391–95 (1952) (9 refs)
Note: T. Urbański & (?). Rychter, CR **208**, 900(1939) found for eleven expls tested by them that logarithmic relationship between the time lag prior to expln and the reciprocal of absolute temperature gives a straight line. Graphs for NC's, MF, ErTeN and EDNA did not give straight lines

Explosive Deflagration. See under Detonative (and Explosive) Combustion or Explosive Deflagration in Vol **4**, p D731-L to D732-L

Explosive or Detonating Compositions of Mediavilla. *Hexamethylenetetramine* (52g) is added to 184ml 100-vol H_2O_2. After agitation at 35° the mixt is cooled to 27° and treated with 50ml 39°Bé HCl. White microscopic crystals are filtered and washed at 27°
Ref: J. Mediavilla, SpanP 203467(1952) & CA **48**, 14210(1954)

Explosive Devices, Register of US Manufacturers. See Expls&Pyrots **5**(4), 1972 (Table covering 2½ pages) and Ibid, **5**(5), 1972 (Table covering ½ page)

Explosive Diode *(One-Way Detonation and Transfer Device).* A unitized device, called an "explosive diode", (US Patent Office Serial No 693358) is seen as a simple improvement over the multi-component, mechanical assemblies normally used for one-way explosive-energy transfer. Typical one-way transfer devices rely upon mechanical functions and percussion primers. Another "assembled components" design used in the industry relies on the precise placement of boosters to assure that a predetermined schrapnel dispersement allows functioning in only one direction. Both have drawbacks that limit their usefulness in the aerospace industry

The explosive diode (See Fig Ex4) consists of a housing into which two cavities are provided to contain a donar charge and an acceptor charge. The position of the donor and acceptor charges within the housing are such that the donor will initiate the acceptor with a shockwave transfer thru the housing. Conversely, the shock generated by the acceptor charge, if detonated first, is dissipated by the geometrical configuration of the cavity in which the explosive is contained, so that any stimulus transmitted to the donor charge is insufficient to effect its initiation

Explosive shocks in an essentially homogeneous medium travel in a well-defined and predictable path either until they dissipate

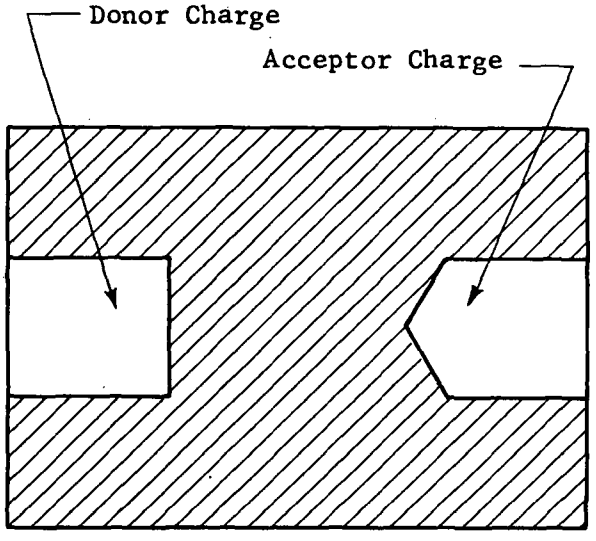


FIG Ex4

their energy (as a function of the impedance of the medium thru which they are traveling), or until they encounter and interface with a medium of a different shock impedance characteristic. Also, when a shock encounters such an interface, it will react in accordance with precise laws of hydrodynamics. The nature of the reaction is generally governed by: (1) the impedance of the associated medium, (2) the geometry or contour of the interface juncture, and (3) other factors associated with the characteristics of the advancing shock front. Thus when the diode's donor is detonated, a shock front is induced into the housing in a direction normal to the flat-bottomed surface of the donor cavity; i.e. toward the acceptor. Upon encountering the angled surface of the cone-shaped acceptor hole, the shock front is transferred in a direction normal to the free surface encountered. The advancing front will essentially converge at a point on the longitudinal axis of explosive material. The result of this converging shock front is an extreme concentration of energy that is sufficient to initiate an explosive having either minimal sensitivity, or a sensitivity such that it might not be initiated by a plane shockwave. This process of shock amplification (or augmentation) permits us to reduce the quantity of explosive in the donor charge or separate the donor and acceptor charges by a greater and less-easily ruptured barrier. Either measure aids in precluding reverse detonation; i.e. detonation of the donor by the acceptor

Geometry is the chief obstacle to reverse detonation. When the advancing shock front encounters the cone at the base of the cavity in which the acceptor charge is placed, the wave is transmitted normal to the surface of that juncture, and spatially dissipated away from the donor charge. It is therefore possible to place equivalent charges in proximity, while maintaining reliable go/no-go performance

The new explosive diode incorporates high reliability, minimum weight, no moving parts, capable of functioning in extreme environments, flexibility in design applications, and good economy

Refs: 1) G.W. Weaver, Explosive Technology, PO Box KK, Fairfield, Calif, 95533 2) G. Cohn, Edit, Expls&Pyrots 1(5), 1968

Explosive Disintegration, Explosive Fracturing or Explosive Shattering. (See also Explosive Unit Operations)

It has been known for many years that certain materials, particularly those of a fibrous nature, can be disintegrated by first saturating them with a gas or vapor at high pressure and then releasing that pressure as rapidly as possible. One of the first to apply this method to practical use was Bancroft (Ref 1). He heated water-soaked crude mica in a pressure-tight cylinder to some desired pressure and then suddenly released it. The expl effect split the thick pieces of mica into thin leaves, suitable for preparing laminated insulation. In 1926, Mason (Ref 2) patented a process for exploding wood chips to prepare a product suitable for direct refining to paper or fiber board. Steam pressures up to 400-600 psi were used. Later patents of the same inventor, listed in Ref 6, improved the method. Pressures as high as 1000 psi were used

Dean and Gross (Refs 3 and 4) carried out extensive investigations on the size reduction of ores by various intermittant expl processes

Godwin (Ref 5) found that coal can be partly pulverized by saturating it with superheated steam under high pressure (1500 psi) and then releasing the pressure by opening a valve

The Germans learned before WWII that if a material with a high internal moisture content is suddenly introduced into a stream of hot gas, a considerable disintegration takes place, caused by generation of steam inside the pores of the material. The **Winterschall-Schmalfeld** process was a commercial application of this property

Refs: 1)G.J. Bancroft, USP 1383370 (1921) (Splitting of crude mica by explosive forces) 2)W.H.Mason, USP 1578609 (1926) (Splitting wood by expln) 3)R.C.Dean & J.Gross, "Explosive Shattering of Minerals", USBur Mines Report of Investigation **RI3118** (1932) 4)IBid, "Crushing and Grinding", USBurMines

Bulleti n No 402 (1938) 5)(?) Godwin, "Coal Pulverization by Internal Explosion", Armour Research Foundation Report August 1939 6)D.Meigs, ChemMetEngrg 48, 122-25 (Feb 1941) (Explosion unit operation of the process industries) (21 refs) 7)J.H.Perry, "Chemical Engineers' Handbook", McGraw-Hill, NY (1950), pp 1166-67; J.I.Yellott, "Explosive Disintegration" 8)Perry, 4th edit (1963), p 8-58 (Expl disintegration)

Explosive Driven Anchor. Accdg to Goodyear Aerospace Corp of Akron, Ohio, 44315, expl driven anchors were proposed to be used to moor a very large balloon. Periodically the balloon must be moved and anchored with extreme rapidity with emphasis on anchoring the balloon securely in the shortest time possible. The expl driven anchor concept appears to be the most expeditious means for this task. The anchor must be capable of being driven into all types of soil and sustain a 1000 pound pull for a period of 48 hours. Fragmentation cannot be tolerated upon actuation of the expl charge

Ref: G.Cohn, Edit, Expls & Pyrotecs 1(11), 1968

Explosive Driver. It is a device for efficiently converting the chemical energy of a HE to the kinetic and internal energy of a gas. The basic element is a thin-walled metal tube, filled with a desired gas, surrounded by a concentric cylinder with HE, such as NMe (Nitromethane). A detonation, initiated at one end of HE, collapses the metal tube as the deton wave front propagates thru the cylinder in much the same manner as toothpaste is squeezed from a tube. The progressive collapse of the tube can be thought of as a piston moving at the deton velocity of the HE. Since ordinary HE's have deton velocities that range from 5 to 9 thousand meters per second, these are the gas velocities that can be achieved. Temperatures resulting from the shock wave can reach 5 to 15 thousand degrees Kelvin, while pressures of 8 to 300 thousand psi are possible. The conversion of chem energy of an HE to the kinetic and internal energy of the gas occurs in a controlled manner to produce specific energy densities in the gas that are significantly higher than those obtainable by more conventional techniques

Potential applications for expl drivers can found in facilities or experiments that require or utilize a high density gas. Ionization, dissociation, radiations, and non-equilibrium within the gas itself can be studied. The gas can be expanded to high velocity and used for investigate hypersonic flow phenomena resulting from its interaction with various aerodynamic models or structures; or it can be employed as an intermediate medium for accelerating other gases or projectiles to high velocity

Refs: 1)"Explosives Drivers", Physics International Co 2700 Merced St, San Leandro, Calif, 94577 2)Gunther Cohn, Edit, Expl & Pyrotcs 3 (2), 1970

Explosive of DuPont Co. Coal Mining expl contd: AN75-90, PETN 7-20, the rest being Na nitrate. Carbonaceous material 3-10% could be added

Ref: E.I.du Pont de Nemours & Co (Inc), Ger P 1130342

Explosive "E". See Explosif E (Swiss) in this vol

Explosive Effect of an Explosive. Accdg to Naoúm, NG (1928), pp 154 & 156, tests like Trauzl or Crusher give a practical measure of the "explosive effect of an explosive" or the work done in actual practice by an expl

Explosive Emulsion. A blasting agent is prepd by emulsifying up to 70% of a liq or meltable hydrocarbon with a liq or meltable expl using up to 5% emulsifier. An example is: NG 50cc, liq paraffin 50cc, oleic acid 1cc & sol Guncotton 1.2g. The common inorganic oxygen carriers, such as AN, may be also incorporated to give higher brisance

Ref: Dr. A. Berthmann et al (to Dynamit Nobel AG, Troisdorf, Bez Köln), Ger P 1152925 (1963), abstracted in Explosivstoffe **1964**, 143-44

Explosive Envelopes & Packages. An active campaign has been conducted by terrorist elements since Dec 1971 to deliver envelope and/or package bombs to selected personnel who are in disagreement with terrorist activities. Initially, packages were mailed from Israel and in July & August 1972, letter bombs were intercepted that had been mailed from Beirut, Lebanon. More recently envelope bombs have been received in several nations, including USA, Canada, England & Germany which were mailed in Amsterdam, Holland and Penang, Malaysia

The basic descriptions of the bombs identified to date are:

a) Envelope bombs varying in size from 3 x 6 inches to 8 x 11 inches. The thickness has varied from 1/8 to 5/16 inches with a weight of approximately 1.7 ounces. Both standard airmail and plain envelopes in a variety of colors have been found. Addresses have been either handwritten or typed on a 1 3/4 inch x 3 1/4 inch white label with red trim or directly on the envelope. It is important to remember that with clandestine devices of this type, the envelope sizes, shapes, colors and postmarks may vary extensively

b) Package bombs recently received have been about 5 x 5 1/2 inches and have weighed approximately 1 pound. Each was packaged as either a gift desk calendar, book or box of candy. The outside package cover was an envelope and most packages were addressed in green ink. These items also could be received in a great variety of sizes, shapes and colors

It should be stressed that all previously described items may be mechanically or electrically initiated to explode when the envelopes or packages are opened, resulting in death or injury to personnel. Should envelopes or packages resembling above descriptions be encountered, avoid handling the items insofar as possible, evacuate personnel from the immediate area, and notify the appropriate Explosive Ordnance Disposal personnel

Ref: Major D.E. Kreul, Disposition Form **2496**, Subject: "Crime Prevention Tips", dated 2 Jan 1973 (Picatinny Arsenal, Dover, NJ)

Explosive Excavation, Instant. The Corps of Engineers is in the process of developing a construction technique called "explosive excavation" which promises to save money, time, and the environment. Rows of 1 to 10 ton explosive charges are buried in a pattern to both break up the material and move it out of the excavation (See Fig Ex5). Controlled blasting techniques minimize fracturing beyond the excavation boundary. Illustration shows arrangement for a 500-foot long railroad cut at Trinidad, Colorado. In several demonstrations, cost was less than

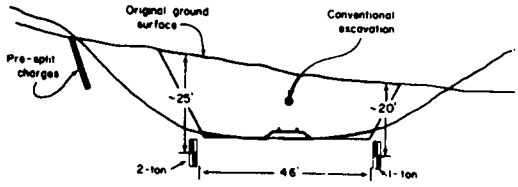
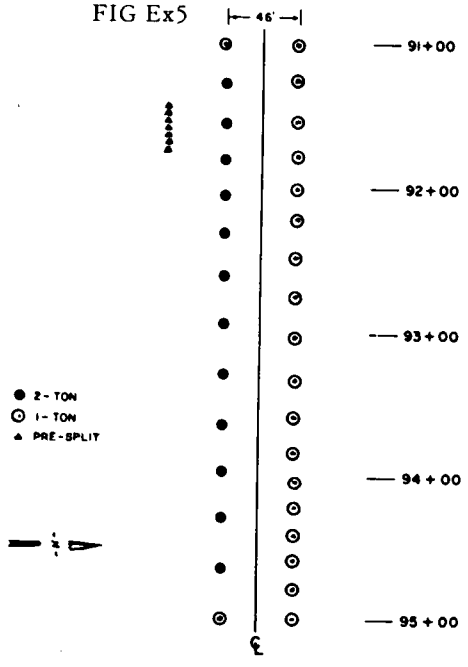


FIG Ex5



conventional earth moving or dredging operations. In underwater detonations, fish kill is said to be no greater than when using conventional methods

Refs: 1) LTC R.L. La Frenz, "Instant Excavation", Army Research & Devel, News Magazine, Vol 13, Nov 4, 1972, pp 28–30
2) G. Cohn, Edit, Expls&Pyrots **6**(4), 1973

Explosive and Incendiary Ammunition. Accdg to reprint available in PicArsnLibrary, Vol 2 of "Small Arms and Small Arms Ammunition" obtained from the Office of Chief of Ordnance, Jan 1946, reported:

"During the period 1920–1941 extended research was conducted by the Ordnance Department to develop small arms explosive bullets. No round was developed which functioned with complete satisfaction in a hot machine gun barrel, the decomposition temperature of all known military explosives is lower than the temperature encountered in the combat firing of small arms machine guns. The nearest approach to a satisfactory explosive cartridge was the TI Pomeroy type

"Explosive bullets were abandoned during WWII due to the limited quantity of explosive which could be carried in a small arms projectile, as well as the effectiveness of incendiary ammunition which resulted in complete aircraft or vehicle destruction

"Throughout the war the popular conception of the laymen was that the army needed an explosive cartridge. Numerous types were submitted for test, none was adopted"

The first expl cartridge was developed by the OrdnDept before WWII. It was **Cartridge, Spotlight, Caliber .50, T1E1**, assembled with a bullet contg a supersensitive nose fuze which included a floating firing pin, centrifugally operated safety segments, a primer and an expl charge of 22 grains of PETN. The cartridge was found to be unsatisfactory for loading in the caliber .50 machine gun because of the sensitivity of the fuze. The T1E1 expl bullet was therefore dropped by the Ordnance Committee in 1937. The Committee recommended, however, that the development of cal .50 HE ammunition be continued to provide a bullet effective on impact with .125-inch dural sheet

In 1939 the Ordn Dept became interested in the **Pomeroy** design which used Dynamite as an expl chge. Upon firing, the rotation of the gun barrel resulted in the centrifuging of the NG from the inert kieselguhr base, thus arming the bullet in flight. The Winchester Repeating Arms Co manufd limited quantities of caliber .30 and cal .50 cartridges based upon Pomeroy design

In Aberdeen Proving Ground Tests in the spring of 1940, it was found that the cal .50 cartridge designated as **Cartridge, Explosive, Caliber .50, T1,** was satisfactory in many respects, except that it could expl in a very hot machine gun barrel. As a result of these tests, the cal .50 cartridge was standardized and 15000 rounds were manufd during 1940 (Its bullet is shown in Fig Ex6). During the same period 3000 rounds of **Cartridge, Explosive, Caliber .30, T1** were manufd (Its bullet is in Fig Ex7)

Meanwhile, development work by the Army Air Forces on incendiary ammunition resulted in decreased emphasis being placed upon the explosive cartridge project. As a result of tests covering a period of nearly two years, recommendation was made that incendiary ammunition be used instead of explosive for all loadings where the targets are aircraft either in flight or on the ground. The test program upon which the foregoing findings were reached included both cal .30 and cal .50 Pomeroy type ammunition. Compositions of incendiary mixts of WWII are given in Vol 2, pp B329 and B330

In the spring of 1943, F.L. Shovic, an employee of the Army Air Forces at Great Falls, Montana, reported that be removing the standard incendiary mixture from the cal .50 M1 bullet and replacing it with a mixture composed of 8 parts K chlorate, 4 p of sulfur, 6 p Mg, 3 p Sb sulfide and 1 p hydrogen-reduced iron powder, the bullet had greater destructive power in firing against aircraft. The tests conducted at Frankford Arsenal showed that the Slovic bullet was not as effective as Bullet, Incendiary, Caliber

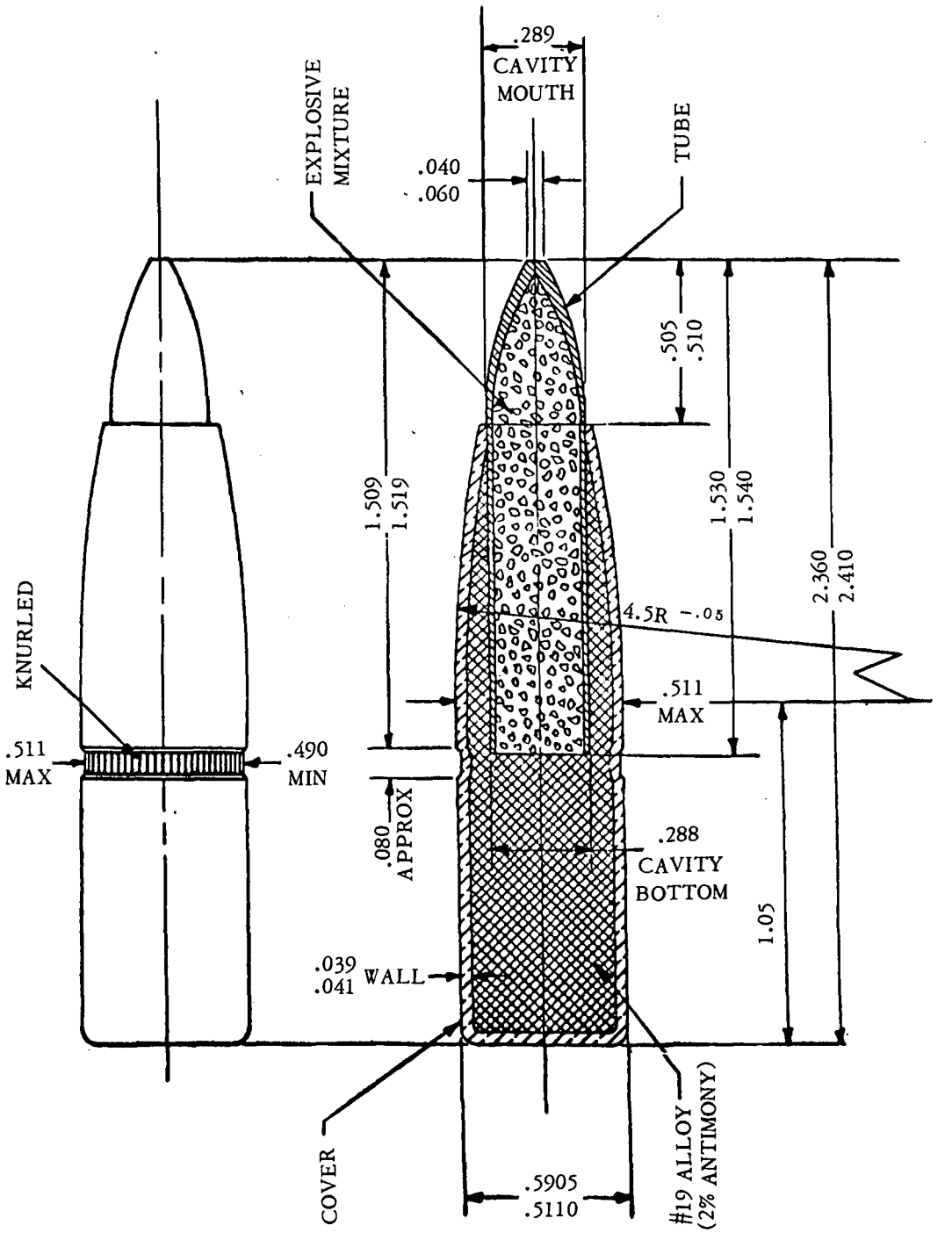


FIG Ex6
BULLET, EXPLOSIVE,
CALIBER .50, T1

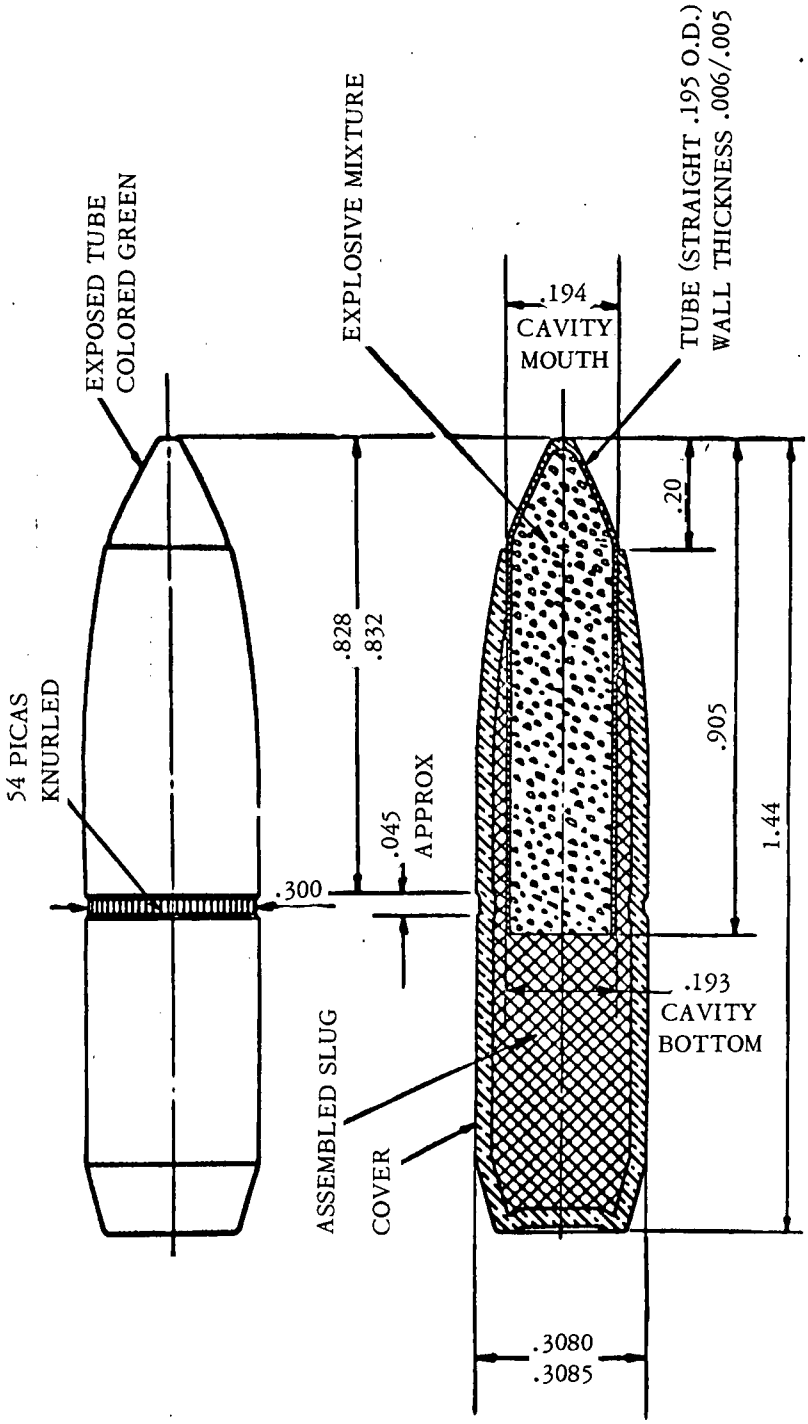


FIG Ex7
BULLET, EXPLOSIVE
CALIBER .30, T1

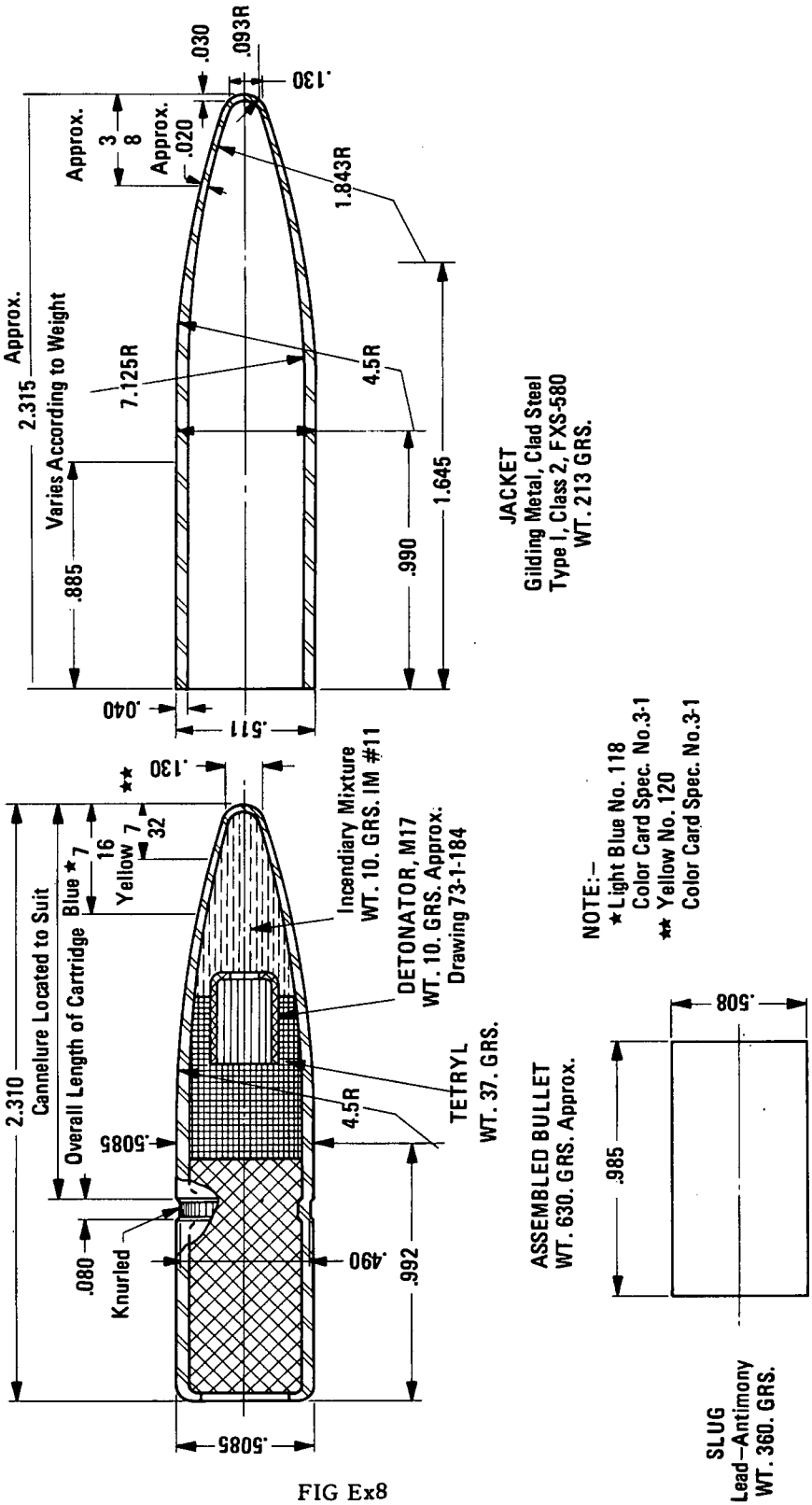
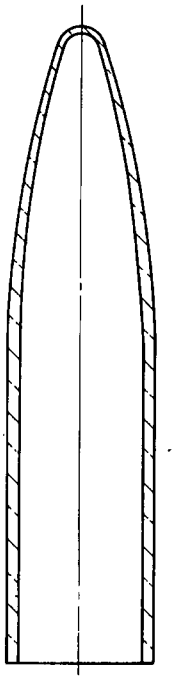
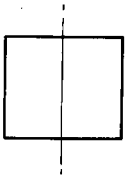


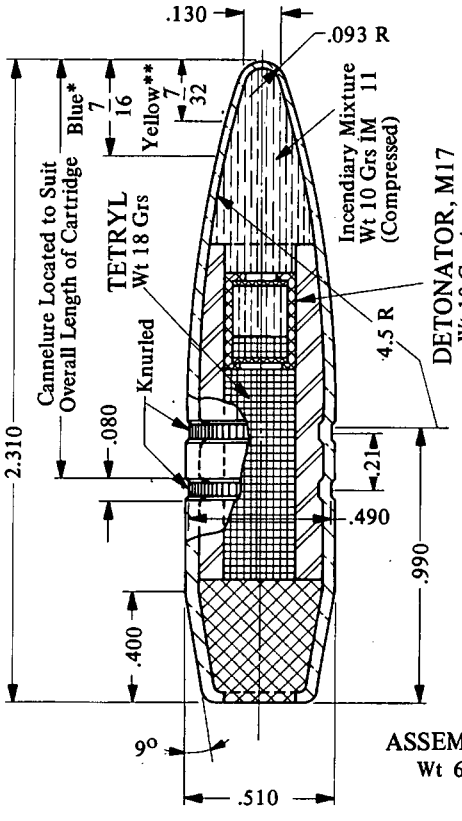
FIG Ex8
BULLET, INCENDIARY, EXPLOSIVE,
CALIBER .50, FA T1



JACKET
Gilding Metal
Wt 235-5 Grs
For Dimensions See
Drawing B174992

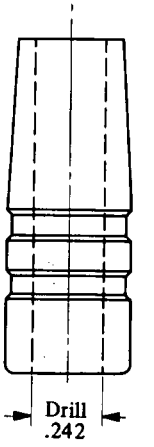


SLUG
Lead–Antimony
Wt 137-4 Grs
For Dimensions See
Drawing A185029



NOTE:-
* Light Blue No 118 Color Card Spec No 3-1
** Yellow No 120 Color Card Spec No 3-1

ASSEMBLED BULLET
Wt 602 Grs Approx



PIN, TUBULAR DOWEL (LARGE)
FXS 535 – Type I
Steel–WDX1112 (S.R.Q. Macro Etch) Class A
Powdered Iron – Class B – Hardened
Steel Tubing – Class C
Finish
Wt 197 Grs Approx
For Other Dimensions See Drawing A299569

FIG Ex9
BULLET, INCENDIARY, EXPLOSIVE,
CALIBER .50, FA T2

.50, M1. It was also shown that Slovic mixture is very sensitive and this was confirmed by extensive tests conducted at Picatinny Arsenal. No further consideration was given to the Slovic bullet

In an effort to develop a more efficient incendiary bullet, a project was initiated before 1944 at Frankford Arsenal to develop bullets combining the properties of HE (high expl) and I (incendiary) type bullets. The following types, belonging to **Cartridge, High-Explosive-Incendiary, Caliber .50, T34** were submitted in 1944 to tests:

Bullet, Incendiary, Explosive, Caliber .50, FA T1, shown in Fig Ex8 consisted of gilding metal clad steel jacket, contg 10 grains of compressed incendiary mixture IM**11, detonator M17, 37 grams of Tetryl added successively and 360 grains Pb-Sb slug; its base was flat; total wt of bullet 628 grains

Bullet, Incendiary, Explosive, Caliber .50, FA T2, shown in Fig Ex9, was manufd by inserting into gilding metal jacket a pin tubular dowel (large), contg .242-inch hole, 10 grains of incendiary (compressed) IM-11, detonator M17, 18 grains of Tetryl added successively and Pb-Sb slug weighing 137 grains; its base was boat-tail; total wt of bullet 600 grains

Both types of bullets were assembled into complete rounds with 227 grains of IMR-4903, AL-2674, as a propellant

Note 1: Incendiary IM-11 consisted of 50/50 Mg-Al Alloy 50 & Barium Nitrate 50%

Note 2: Propellant IMR-4903, AL-2674 is presumed to have consisted of NC (13.5-13.25% N) 98.3, K_2SO_4 1 & DPhA 0.7%

Following were ballistics for above rounds:

Round	Velocity at 78 feet	Pressure
FA T1	2909 fps	49220 psi
FA T2	2918 fps	49060 psi

Preliminary firings indicated that the bullets did not withstand "cook-off" tests. More detailed tests conducted at the Ordnance Research and Development Center gave the following results:

When a round contg FA T2 bullet was placed in a hot machine gun barrel after 100-round burst, no premature ignition or expln of the bullet took place

When an identical round was placed in the barrel after 125-round burst, premature expln of the bullet occurred with bulging of the barrel

The bullet placed after 150-round burst detonated with force splitting the barrel and bulging the chamber

Similar results were obtd with rounds contg FA T1 bullet

The above "cook-off" tests proved that both types of ammunition are not suitable for use in machine guns

Similar unsatisfactory "cook-off' prematures were obtd with Caliber .50 Explosive Bullet developed by the Hunter Manufacturing Co and submitted in 1943 to tests at Aberdeen Proving Ground. The bullet had a firing pin, a LA detonator, an arming wire in the nose, and an expl chge of 15 grains of Tetryl

Explosive Fabrication of Metals. This term includes **explosive forming, –forging, –sizing –welding, –compaction,** and **–conditioning.** Other processes of minor interest are embossing, shearing, flanging, joggling, threading, punching, pressing and extrusion

Accdg to V. Philipchuk (Ref 7), in conventional process of metal fabrication the pressure is usually supplied by a hydraulic press, while in expl fabrication the pressure is supplied by expln of a calculated charge of expl. The difference betw the two techniques is the pressure-time work curves, which, essentially, show a major difference in stress-strain applications

Fig Ex10 shows the difference in the stress rates in conventional and expl forming. In expl forming the metal moves rapidly thru its elastic range and into its plastic range, producing permanent set of shape within microseconds. A complete understanding of the action taking place in the metal under high stress-strain rates is not known and here one can only theorize. Practically all metal-fabricating processes require forces of vary-

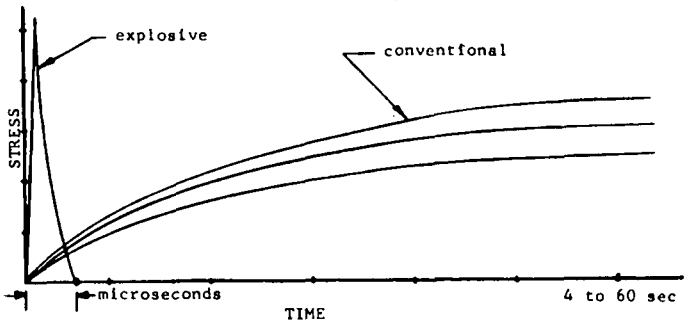


FIG Ex10

Conventional versus explosive stress rates

ing degrees and application. The expl forces used vary with the amt of expl, its type, and the location of pressure monitor from the expl chge. With an improper combination, these forces can either not affect the metal part or shatter (fracture) it. This fracturing usually occurs when the stress duration has been applied for too long a period and this might take place before the normal elongation point of the metal is reached

As in other metal fabricating techniques, the temp of the metal is of importance, although most of the work to date has been conducted at ambient temp

True expl forming does not utilize the high range of expl pressures on the metal part itself but transfers this pressure thru a medium which essentially dampens the extremely high pressures and provides the desired distribution of forces to the metal part for forming. The metal forming takes place when these forces strike the metal with sufficient energy to move it thru the elastic and into plastic range of the metal or alloy. Generally, one should envision a pressure pulse striking a metal part with sufficient force to deform it and yet with insufficient force to structure it (Ref 7, pp 1–2)

Accdg to Ref 10, the use of expls to do construction work is not new. About 1888 Amer scientist C.E. Munroe (1849–1938) used expl charges to engrave names and designs on iron plates by procedure in use at the present time. As a result of his work and of the work of German scientist E. Neumann, the process known as the "shaped charge effect" was developed. It is described in Vol 4 of Encycl, pp D442-R to D457-R, under DETONATION, MUNROE-NEUMANN EFFECT

Prior to 1900, German, American and British engineers were awarded patents for expl forming methods, but the technique lay dormant until about 1954 when interest in it revived. The advent of the missile age with its large and complex parts, high strength materials, close tolerances, and relatively low production volumes appears to have provided the greatest impetus to the method, although the Moore Co of Marceline, Missouri, was one of the first to recognize the benefits of this method for commercial production operations

Very important work, beginning prior to 1958, was done at the National Northern Plant, West Hanover, Mass under the direction of Philipchuk (Refs 4 & 7). There were manufd small and large parts for aeroplanes and jet-motors. For example, the jet-motor housing shown in Fig Ex11 was explosively formed

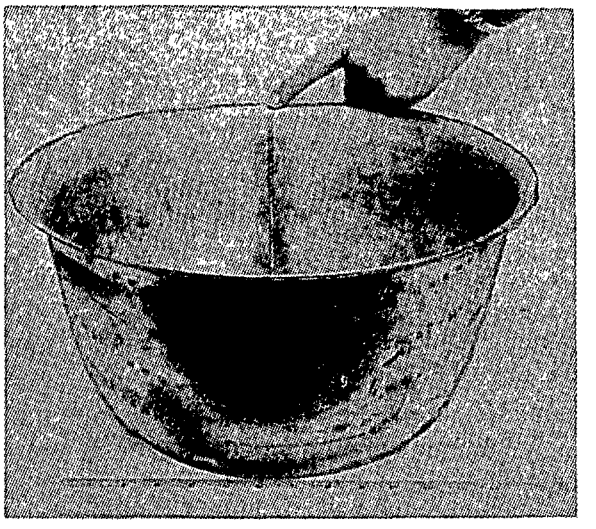


FIG Ex11

Jet-motor housing, explosively formed

Accdg to Philipchuk (Ref 7, p 2), the methods used for the various fabricating processes depend upon the process: i.e. forming, forging, welding, etc. In general, the principal method consists of immersing the designed expl charge into water, with the water being in contact with one side of

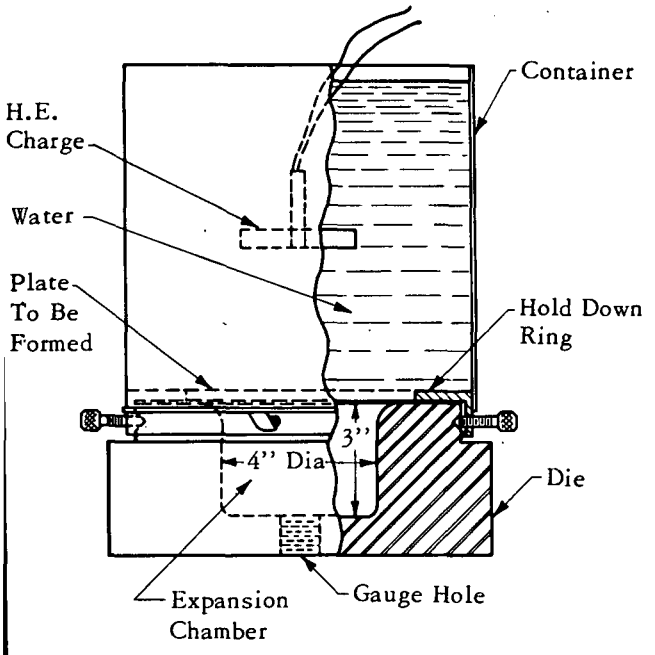


FIG Ex12

Basic forming method

the metal part (Fig Ex12). A female die having the desired finished contour is behind the metal part. Depending upon the starting and final shapes, the necessity for a vacuum betw the die and the part is determined. The expl chge is detonated, transmits the pressure pulse to the metal part, and forces it into the shape of the female die

A simple arrangement, shown in Fig Ex13, was published in the Fortune Magazine for Sept 1961 (Ref 9). The device consists of a liquid contg tank, female die, vacuum pump and an expl with some means of detonation. The liquid (such as water) served as a medium for the transferral of the shock waves (which supply the major portion of the energy) and gas pressure generated by the detonation of the expl. A vacuum pump is required to evacuate the die cavity to reduce forming resistance and to eliminate the burning of the die side of the material and the die. This burning is a result of the excessive temps (as high as 10000°F) generated by the compression of the gas entrapped in the die cavity. It was necessary in some cases to draw a vacuum of 29.5 inches Hg in order to prevent the autoignition of the die lubricant which occurred at higher absolute pressures. This autoignition can create spring-back and dents in the part being formed

The transfer media can be, besides water: hydraulic oils, molten salts, molten alloys, rubber, glass, sand, air or inert gas. The expl may be PETN, RDX, TNT, NG, 30% Ammonia Gelatin or NGu (Nitroguanidine) and may be used in the form of a powder, sheets, liquid, cords, pellets or cylinders. In general, any expl that is homogeneous and can be handled safely may be used (Ref 10)

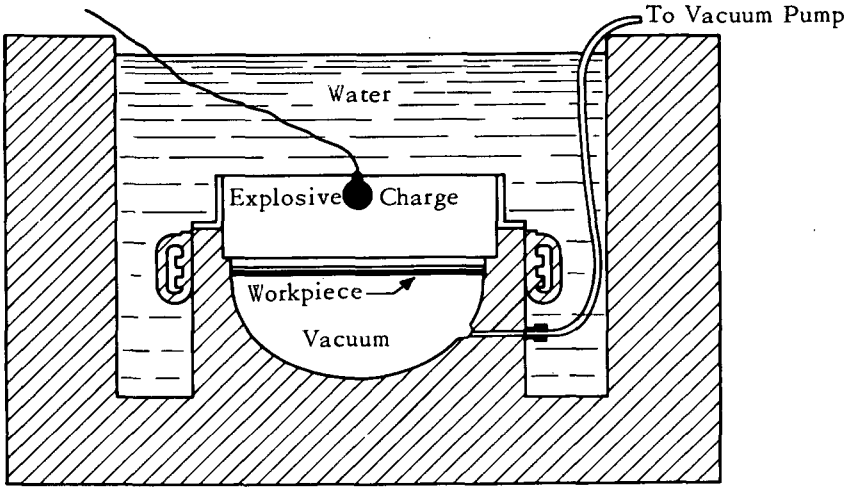


FIG Ex13

The metals and alloys that have been under test at National Northern were before the publication of Ref 7 the following: plain carbon steels, alloy steels, stainless steels; Ni, Al, Mg, Ti & Cu alloys; Zircalloy-2; Ta, U, Ber, Niobium (Columbium), W (Wolfram or Tungsten) and Mo. They have been in either one or more of the following starting forms: sheet, plate, cylinders, cones, powders and pellets

Weights of parts formed have ranged from a few grams to over 2 tons

The methods for forging, welding, compaction and conditioning require preparation of the metal and die surfaces prior to the operation. Each of these operations requires higher prr ssure forces than those needed for expl forming of sheet material. Most of the operations are such that the fabricating process can be set indoors. Extremely large parts, or those requiring great quantities of expls, are processed out-of-doors to prevent residual and reflected shock or blast fronts from damaging the buildings (Ref 7, p 3)

Explosively forged materials manufd at National Northern exhibited physical properties above minimum conventional requirements and are considered of good quality (Ref 7, p 5)

Explosively compacted materials, such as Ti and steel, have produced small pellets, ca 1 x 1 inch, having densities of over 95% of theoretical without a sintered operation. It is there that a new branch of powder metallurgy, i.e. high pressure in place of high temp, and a combination of both, is under way (Ref 7, p 5)

Explosive conditioning mentioned in Ref 7 is not described

Explosive extrusion has been investigated by the Air Materiel Command of the US Air Force but it is not described in Ref 7

Explosive welding, mentioned in Ref 7, pp 3 & 5 is described as a separate item

Explosive forming is used for manuf parts which cannot be made by other methods (See also Explosive Metallurgy; Explosive Metalworking, High-Velocity; Explosive Press; and Explosive Welding)

Refs for Explosive Fabrication of Metals:
1) Anon, Steel **143**(8), 82-6(1958) (Pressing and drilling holes by using shaped charges) 2) Anon, Materials and Design Engineering **47**(1), 168-72(1958) (Explosive forming, shaping and punching of metals) 3) Anon, Product Engineering **30**, April 20, 1958, p 26 (Explosive forming) 4) V. Philipchuk, "Explosive Forming of Metals in National Northern", AP&CC, August 1958 4a) Anon, Steel **144**(3), p 62(1959) (Why explosive forming works) 5) Anon, Machinist **103**(12), 127-38(1959) 6) H.T. Hall, "Ultrahigh Pressures", Scientific American, November 1959 7) V. Philipchuk, "Metal Fabrication by Explosives", AmerSoc of MechEngrs, ASME Paper No **60-MD-4**, May 1960, pp 1-5 8) R.W. Heinemann et al, "Explosive Forming of Explosives", Feltman Res & Engrg Labs, PicArsn, TechRept **2685**, June 1960, Project TW-120, Project 504-03-61 9)Anon, Fortune Magazine, September 1961 (Explosive Forming) 10)Anon, "Review of Explosive (Chemical)Forming", USArmy Production Equipment Agency, Manufacturing Technology Division, Roch Island Arsenal, Illinois, April 1965 (164 refs) 11)K.Kiyota, Kogyo Kayaku Kyokaishi **26** (1), 35-9 (1965); CA **64**, 3274 (1966)(Explosive forming technology. A review) 12)A.H.Holtzman & G.R.Cowan, Ordn **50**, 536 March & Apr 1966 (Explosive Forming) 13)J.Pearson, NAVORD Rept **7033**(NOTS TP2421), China Lake, Calif 1966 (Expl working of metals) 14)G.Cohn Edit, Expls & Pyrots **4** (7), 1971 (Expls forming for manuf metal plate dentures)

Addnl Refs on Explosive Fabrication of Metals. A) I.I. Islamoff, "Metal-Forming Techniques", NASA, **SP-5017**(May 1965) B) M.C. Noland et al, "High Velocity Metalworking, A Survey", NASA **SP-5062**(1967)

Explosive, Flexible of Kegler. It may be prepd by mixing an expl with an elastometer (depolymerized natural or synthetic rubber) and a vulcanizing agent. Vulcanization is carried out simultaneously with formation of the product. For example, 500g dry PETN of particle size <100μ was mixed with 200g liquid depolymerized natural rubber(such as

Lorival-R), having viscosity of about 5000 cp, to give viscous mass which was subjecting to flatting and 200g of PETN added. When the mixture was homogeneous, 5g Colloidal S, 5g ZnO, 2g Vulcacit P, and 3g antioxidant 4010 were added, and the resulting mass was converted to plates on a 2 cylinder colander

Ref: W.Kegler to Institute Franco-Allemand de Recherches de Saint-Louis, FrP 1308664 (1965); Swiss Appl June 19, 1963 & CA **64** 3276-77 (1966)

Explosive Force. Its definition is given in Vol **4** of Encycl, pp D391-R & D392-L under Detonation (and Explosion) Impetus and Available Energy. Haid & Koenen (Ref) described a modification of Kast's Apparatus, known as Brisance Meter of Kast, shown on p C493 of Vol **3**, which they used for measuring expl force. Comparative values obtd by crushing Cu cylinders were TNT 10.1, DNBz 6.2, PA 12.2, Tetryl, 14.0, PETN 17.0 & MF 18.6
Ref: A.Haid & H.Koenen, SS **28**, 369-72 (1933) & **29**, 11-14 & 13-17 (1934) & CA **28**, 2907 (1934)

Explosive Forging. Mentioned but not described under Explosive Fabrication of Metals

Explosive Forming. See under Explosive Fabrication of metals

Explosive Fracturing or Shattering. See Explosive Disintegration

Explosive Fracturing for Well Stimulation. Accdg to Refs the USBurMines in cooperation with Sun Oil Co investigated the application of expl fracturing for stimulating natural gas recovery. Two tests conducted in the shallow Buzzard Sand formation in Osage County, Oklahoma, demonstrated that desensitized NG can be safely loaded, displaced and detonated in hydraulically induced fractures both in open-hole intervals and thru casing perforations
Refs; 1)W.D.Howell & T.J.Clare, "Case History-Explosive Fracturing for Well Stimulation", Proc Spring Meeting Mid-Continent District, Div-Production, API, Wichita, Kansas, April 8-10, 1970, pp 58-63 2)G.Cohn, Edit, Expls & Pyrots **5**(6), 1972 (Abstract)

Explosive Gas Gun. The gun consists of a steel pressure tube containing helium gas and a projectile. The tube is surrounded by a high expl (Nitromethane). The gun is operated by detonating the HE. The detonating front propagates along the steel tube causing it to collapse. The collapsing wall of the tube acts like a piston traveling at the detonation velocity of the HE. This motion sends a shock wave into the helium gas which accelerates the gas to the detonation velocity of the HE. Projectiles weighing up to 100 grams can be ejected at a velocity of approx 18000 ft/sec. The proposed use of the gun is to simulate condition of re-entry of space capsules. The tentative parameters of such a gun are total length 15 ft, barrel length 4 ft, projectile dia 2.5 inches, projectile length 1.5 inches, projectile wt 101 gms, helium gas 216 g, Nitromethane 143 lbs
Ref: D.Mumma, "Explosive Gas Gun for Re-Entry Simulation", Physics International Company Report **PIFR-024** (1965), San Leandro, Calif

Explosive Gases. See Vol **4** of Encycl, Tables on pp D352, D356 & D360
H.T.Titman & A.Wynn discussed in Rev Ind Minérale **36**, 50-61 (1955) & CA **49**, 9729(1955) the ignition of gaseous mixtures by friction

Explosive Generators are devices for converting energy of explosions into magnetic field energy. They are systems with variable inductance in which the moving conductors, accelerated by an expln, work counter to the ponderomotive force of the magnetic field and thus cause an increase in the energy of the field. Two types of generators were prepd and tested in Russia: 1)a two-bar generator and 2)a coaxial generator. Their construction and results of tests are described in Ref. In

one successful experiment, the efficiency of conversion was 12-14%
Ref: E.I.Bichenkov, Dokl.AkadNauk (Russia) **174**(4), 779-82 (1967) & CA **67**, 6253 (1967)

Explosive High-Velocity Metalworking. See Explosive Metalworking, High-Velocity

Explosive of Holt and Hispano Suiza SA. It was prepd by mixing gelatinized NC with 10-60% of HE (such as RDX,TNT,etc) and oxidizing salts (such as nitrates, chlorates and perchlorates). The solids had grain sizes of 0.1-2.0mm and were homogeneously distributed in NC jelly
Ref: E.von Holt & Hispano-Suiza SA, Tanger, GerP 1010887 (1957) & CA **54**, 8087 (1960)

Explosive Initiation Evaluation. A model described by Evans, is derived based on heat-transfer considerations. It indicates performance of an expl charge in contact with a thermal bridge. The result gives bridge temperature as a function of time, current, and the physical properties of the bridge and expl
Refs: 1)John Evans "Explosive Initiation Evaluation", Instruments and Control Systems **42**(10), 140-41 (1969) 2)G.Cohn, Edit,Expl & Pyrots **3**(4), 1970

Explosive of Iroda. An expl compn claimed to be as powerful as Tetryl or RDX, was prepd by thoroughly blending a pulverized mixture of AN and/or K(or Na) nitrate with Al and a jelly consisting of NC,NG in volatile solvent (such as Methanol). After pressing to the desired density (1.4 to 1.6) and grain size, the solvent was allowed to evaporate
Ref: A.G.Iruda, BelgP 613336 (1963) & Kemixon Reporter, Barcelona **10**, p 286, March 1963

Explosive Lead Salts, Formation of During Manufacture of TNT. Supporting evidence on the formation of Pb salts is given based on studies on the mechanism of purification of TNT by Na_2SO_3 washing
Ref: H.Muraour, MP **36**, 37-39 (1954) & CA **49**, 16435 (1955)

Explosive Linear Charges. See under Explosive Streamers

Explosively Actuated Tools. Under this heading A.H.Oldham, Army Ordn **18**, 361-6, (1938), reviewed some of the patents relating to the operation of tools and other mech. devices actuated by means of expls. Among these tools are cable cutters, cattle killers, circuit breakers, engine starters, joint making apparatus, log-splitting tools, pile drivers, pin driving apparatus, rail bonds, riveters, explosively operated hammers, ship salvage and well-casing tools

Explosive Limits and Flash Point-Derivation of Empirical Equations for the Lower Explosives. A summation of exygen demand for each of the atoms in the fuel molecule gives a value which, combined with the boiling point gives the flash point, and which combined with the reaction stoichiometry gives the lower expln limit. Some oxygen demand values are C+4, H+1, S+4, O−2
Ref: (R). Oehley, "Ableitung empirischer Gleichungen für die untere Explosionsgrenze und den Flammpunkt", Explosivstoffe **1956**, 93

"Explosive Metallurgy" is the term coined by the authors of Ref to embrace the techniques, sciences and phenomena involved in the application of expls to the processing of metals. The term includes items described in this Vol, namely "Explosive Fabrication of Metals," "Explosive Forging", "Explosive Forming", "Explosive Press" and "Explosive Welding" (See also Explosive Metalworking)
Ref: H.P.Tardif & W.H. Erickson, "Explosive Metallurgy", Canadian Mining & Metalurgical Bulletin, June 1958

Explosive Metalworking, High Velocity. Noland et al (Ref 1) included in a well-illustrated 79-page chapter on explosive metalworking the components of explosive processing systems, metalworking facilities, controls, tube forming, sheet metal and plate forming, dishes and domes, hardening and forging, welding and powder compaction. Shorter, but equally well done chapters describe electromagnetic, electro-hydraulic and pneumatic-mechanical metal-working. Authors also give background on die design for sheet metal forming and forging, and on material behavior at high strain rates. Many advantages of the method are cited

As an example "Explosive sizing of cones or cylinders" is shown in Fig Ex 14 (See also Explosive Metallurgy, Explosive Fabrication of Metals, Explosive Forming and Explosive Welding)

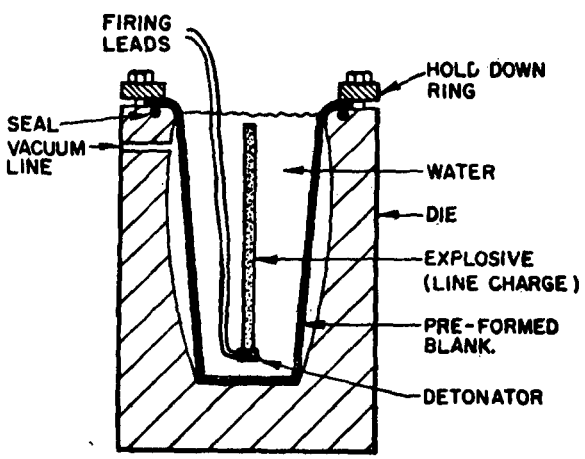


FIG Ex14
EXPLOSIVE SIZING OF CONES OR CYLINDERS

Refs: 1)M.C.Noland et al, "High-Velocity Metalworking, A Survey", NASA **SP-5062**, US Govt Ptg Off, Washington, DC, 20402 (1967), 183+ IX pp, 191 refs. Available from Supt. of Documents 2)G.Cohn, Edit, Expls & Pyrots **1** (6), 1968 (Abstract)

Explosive Mixture, French. See Frantsuzskaya Smes' (Russian)

Explosive Mixture, French and Spanish. See Ref and under individual name of expl
Ref: Société d'Explosifs et de Produits Chimiques, Spain P 236626 (1962) & Kemixon Reporter (Barcelona), Jan **1963**, Item 11-17440

Explosive Mixture of Methanol-Water-Magnesium (or Aluminum). On the basis of theoretical calcns of heat of evolution, the author concludes that mixts of Mg or Al with water or alcs are potentially more powerful expls than the usual military materials, with MeOH-Mg giving max gas evolution. The experiments were conducted in bombs or lead enclosures with Tetryl detonators to set off the mixt of powd metal & liq. All mixts were shown to be powerful expls, with water-Mg being the most sensitive to shock, while water-Al & MeOH-Mg were less sensitive and reqd a booster detonator. Photographs of the exploded vessels & bombs are shown for this new type of expl compns
Ref: A.A.Shidlovskii, ZhurPriklKhimii **19**, 371-78 (1946) & CA **41**,1105 (1947)

Explosive Mixtures. T.Urbański (Ref) reviews properties of numerous expl mixtures, described by previous investigators and also prepd by him and collaborators. The title of his paper is "Sur quelque régularities observées dans les proprietés de certain mélanges explosifs"
Ref: T.Urbański, MAF **20**, 237-63 (1946) (55 refs)[The properties detd include ignition (or expln) temperature, impact sensitivity, Trauzl Test and detonation velocity]

Explosive Mixtures, Detonability of. A study of detonability of liquid binary and ternary mixts of NMe(Nitro methane), hydrazine and methanol showed that hydrazine strongly sensitizes NMe and NMe-methanol mixts to de-

tonation. However, the binary and ternary mixts were insensitive in the drop-weight impact tests. Theoretical calcns yielded reasonable estimates of the energy content of mixts
Refs: 1)D.R.Forshey et al, Explosivstoffe **17**(6), 125-29 (1969) 2) G.Cohn, Edit, Expls & Pyrots **3** (2), 1970 (Abstract)

Explosive Mixtures Detonation of. See Detonation of Explosive Mixtures in Vol **4** of Encyl, p D346-L & R

Explosive Mixtures of Gases, Flame Propagation in. A system of differential equations of flame propagation is set up and discussed. The energy equation reduces to the form of Abel's differential equation of the 2nd type. A simple numerical soln is developed. General theory developed is check by expts with expln flame of O_3 and O. for which complete and reliable kinetic data are available. General theory is applied to mixts of Methane and Nitrogen air, helium air, and argon air. It was found to be applicable only when dissociation of the end products is negligible. Temp distribution in the flame zone is calcd on the well-founded basis of const. enthalpy
Ref: R. Sandri, Can J Chem **34**,313-23 (1956) & CA **50**, 12481-2 (1956)

Explosive Mixtures of High Density. A method of prepn, patented by Cook & Davis, involves subjecting two solid chem compds, at least one of which is a nitrated org compd (such as a mixt of TNT, An and (other compds), to a temperature at which at least a portion of one of the compds is in molten condition. To this molten mass is added a dispersing agent (such as a Na alkyl naphthalene sulfonate or other suitable org sulfonate or fatty acid soap) and then, after thorough blending, the mass is cooled and solidified. Several examples with details are given
Ref: M.A.Cook & C.O.Davis (To E.I.duPont de Nemours & Co), USP 2353147 (1944) & CA **38**, 6098 (1944)

Explosive Mixtures of Koch. They are prepd by coating a high-melting expl with a low-melting one. The latter is added to a strongly agitated suspension in water of the higher-melting expl. Then the mixt is cooled until the low-melting expl solidifies
Ref: G.Hoch to Wasag-Chemie AG, Ger P1038470 (1958) & CA **54**, 16832-33 (1954)

Explosive Mixtures and Phlegmatized Explosives, Physical Methods for Analyzing the Components of. Discussed in refs
Refs: 1)Author, MP **36**, 133-62 (1954) 2) P. Aubertein, MP **38**, 29-37 (1956)

Explosive Mixtures of Streng and Kirschenbaum. High-explosive mixtures contg finely divided metals (such as Al) and an aqueous soln of an oxidizer (such as AN or a perchlorate. Thus 30 parts 60-Mesh dry NH_4NO_3 was dissolved in 28 parts water and resulting soln added with constant stirring to 42 parts of a mixt of equal parts granular 400-Mesh and flake 100-Mesh Al to give suspension with a rate of detn of 4000-200 m/sec when initiated by a No. 6 cap. Dispersing, emulsifying, and wetting agents may be added
Ref: A.G Streng & A.D. Kirschenbaum, USP 2836484 (1958) & CA **52**, 14172 (1958)

Explosive N (Methylite 20 & 25). Expl compns consisting of NG/dimethylphthalate/stabilizer (not specified). The NG used was a commercial grade contg ethylene glycol 25, Ethyleneglycol Dinitrate 30 or Diglycerin Tetranitrate 30% to depress its freezing point. Props of Explosive N are as follows:
Methylite 20 is NG/dimethylphthalate/stabilizer 80/19.2/0-8%. Its fr p -30°, impact sens 49 (TNT=100 cm) & bullet sensitivity-not detonated by .50 cal bullet; initiated by ES cap & booster; and velocity of deton 7000-7300 m/sec at d 1.48 g/cc.
Methylite 25 is NG/dimethylphthalate/stabilizer 75/24.25/0.75%. Its fr p -30°, impact sens 56(TNT=100 cm) & bullet sensitivity-not detonated by .50 cal bullet; initiated by ES cap & booster-and velocity of deton 7000-7300 m/sec at d 1.47 g/cc

Both expls are stable in storage at 80 to 120° F. They are used as hose fillers for mine field clearance
Ref: P.C.Keenan & D. Pipes, NAVORD Rept **87-46**, Table of Military High Explosives, "Navy Dept, Bureau of Ordnance, Washington, DC (July 1946), Part 6. Liquid Explosives

Explosiveness of Ammonia-Oxygen Mixtures, Danger of. A dry mixture of ammonia and air is capable of detonating at 0° over the range 15.5 - 27.2% ammonia, by vol, and at 100° over the range 14.5 - 29.5%. In moist air, however, the danger decreases with increasing temperature; at 0° the detonation range is 15.5 - 27%, at 40° it is 20-25%, and over 45° there is no tendency to explode. The stoichiometric mixture is 22% ammonia, 78% air, and has a detn vel of 0.5 m/sec. At 18° the detonation range for ammonia/oxygen is 16.7-79 vol % ammonia; by 100° the lower limit has fallen to 16% ammonia
Ref: E.Banik, Explosivstoffe **1957**, 29-32 and **1958**, 145-47 (7 refs)

Explosiveness and Constitution. According to Majrich, the explosiveness depends not only on the nature of the carbon chain, but also on other groups. For instance the aldehyde, Keto and carboxylic groups lower the brisance, sensitiveness and stability, especially in nitric esters and the stability seems to increase with increasing mol wt
Ref: A. Majrich, Chem Obzor **7**, 104-8, 117-119 (1932) & CA **26**, 5209 (1937)

Explosive Nitric Esters, Liquid. Spout wash water formed in the mfg of liquid expl nitric esters is treated with a non expl aromatic nitro hydrocarbon e.g. 0-Nitro toluene (I), to extract the expl esters and render the wash water safe for disposal. Wash water from a Nitro glycerine (II) plant contg 0.35% II was treated at a rate of 25000 lbs/8 hrs with 1000 pounds of (I) which was continuously recycled so as to give a 2:1 wash water:I vol ratio
Ref: W.G.Allan et al, Brit P734523 (1955) & CA **50**, 2177 (1956)

Explosive or Blasting Oil. A laymen's term for oily expls NG, NGe, etc used in Dynamites and other Blasting expls
Refs: 1)Naoúm, NG (1928), p 4 2)CondChem Dict (1961), 481-R

Explosive Operated Valve. A device to seal the sample containers of the Cane Sequenced Sampler used by the Oak Ridge National Laboraties to obtain gas samples of underground detonations of nuclear explns, (Project Gnome). The valve design is based upon the use of aluminum tubing, 0.5 inch O.D. and 0.065 inch wall. The explosive load consists of 17½ inches of 30 gr/ft. PETN Primacord wound in five tight turns-about the tubing. A detonator is attached. For confinement of the explosion, a shell of mild steel tubing with a neoprene liner is slipped over the expl charge. A seal is obtained that has all appearances of a weld
Ref: W.E.Fogg, F.A. Memorandum Rept **M63-12-1**, An Explosive-operated valve (1962) Pitman-Dunn Laboratories, Philadelphia, Pa

Explosive Output Testing. Under this title V.J. Menichelli briefly describes in Ordn **48**, 663-66 (1964), the following tests for detonators; Trauzl Test, Sand Bomb Test, Steel Dent Test and Lead Disk Test

See also the following reports describing expl output testing fixtures & procedures:
a) M.L.Schimmel & V.W.Drexelius, "Measurement of Explosive Output", Proceedings of the Fifth Symposium on Electroexplosive Devices, Franklin Institute, Philadelphia, Pa (June 1967)

b) M.G.Kelley, "A Radiant Energy Technique to Measure Explosive Output", Ibid

c) E.R.Lake, "Percussion Primers, Design Requirements", McDonnell Douglas Corp Rept **MDC A0514**, St. Louis, Missouri (June 1970), p 7 (output characteristics)

d)R.K.Warner, "Measuring Output, Safety and Reliability of Explosive Components", Harry Diamond Laboratories Tech Memo **TM-71-31**, Washington, D C (Nov 1971)

e) W.E. Voreck, "Detonator Output Measurement", Minutes of Department of Defense, Fuze Engineering Standardization Working Group, Picatinny Arsenal, Dover, N.J. (April 1973), Appendix 9

NOTE: See also Vol 4 of Encycl, pp D1084ff (Output Tests)

Explosive Papers or Pyropapers (Papiers explosibles or Papier fulminants in French; Papel explosivo, in Span). Under these names are known absorbent (such as filter) papers impregnated by solns of various substances and then dried. They served as basis of several propellants and expls

Daniel (Ref 1, p 599), under the title *Papier explsible* describes an expl prepd by impregnating filter paper with aqueous suspension of saltpeter 41.66, K chlorate 41.66, charcoal or coal dust 8.34 & sawdust 8.34%, followed by drying; some glue was dissolved in water to make the suspension adherent to the paper

Reichen was the first to utilize explosive paper as a basis for expls. He patented in 1865 in England an expl which he called *Melland (Papier-poudre)*, instead of calling it *Reichen poudre* (Ref 1, p 436)

One of the expls utilizing "explosive papers", *Dynamogene*, described in Ref 1, p 231-32, is also described in Vol 4 of Encycl, p D1740-R

Other expls and propellants based on expl paper are: *Gelbite* (described under Emmens in Ref 1, p 257); *Hochstätter* (Ref 1, p 376; *Mèche lenté* (Ref 1, p 433; *Peley (Papier explosible)* Ref 1, p 602; *Prentice* (Ref 1, p 651); *Pyropapier* (Ref 1, p 665); *Reichen* (See Melland, Ref 1, p 436); *Spiralite* (Ref 1, p 735); and *Unionite* (Ref 1, p 779)

All above substances will be described later

Pérez Ara (Ref 2), under the name *Papel explosivo de Pelez* describes an expl prepd by wetting an ordinary blotting paper in aqueous syrupy suspension contg: K clorate 45.5, K ferrocyanide 11.5, Na chloride 24.5, charcoal 11.5 & starch 7.0%. This compn is similar to that of *Peley (Papier explosible)* given by Daniel (Ref 1, p 602) as: K chlorate 47.50, K ferrocyanide 12.42, Na chloride 24.82, charcoal 8.17 & starch 7.09%

Refs: 1) Daniel (1902), 231-32, 257, 376, 433, 436, 599, 602, 651, 665, 735 & 779 2) Pérez Ara (1945), 213

Explosive PB-RDX. See Explosive Composition PB-RDX

Explosive Pentolite. See Explosive Compositions Pentolites

Explosive Performance, Comparison of Two Methods for Its Evaluation. Until the middle of 1950's, the choice of expls for blasting a particular rock was made on the basis of the following methods: a) Trial and error gained from previous experience in similar rocks; b) Calculation of meaningful performance parameters of expls from their chemical compns by means of laws of thermochemistry or thermodynamics; or c) Laboratory determination of expl characteristics

Although good results have been obtd with expls chosen on the basis of these methods, it was not unusual to find quite different expls being used in very similar rocks

In 1956, Duvall & Atchison of USBurMines (Ref 1) developed an experimental technique for making quantitative measurements of the strain produced in a solid rock medium by the deton of an expl chge. This technique has been used to study the physical process of rock breakage. The studies have shown that the characteristics of the strain pulse produced in a rock by an expl determine the effectiveness in breaking rock. Thus, the strain-measuring technique provides a direct method for measuring the relative effectiveness of different expls (Refs 1, 3 & 7)

The Spencer Chemical Co, Kansas City, Missouri, thru a contract with the Armour Research Foundation, was working on an underwater testing method for evaluating the relative effectiveness of different expls by measuring the pressure produced in the water by the de-

tonation of small expl chges (Ref 6). The method was simpler and cheaper than making strain measurements in rocks and provided conditions somewhat closer to deton in rock medium than many other types of performance tests. However, the differences in conditions were still great enough to raise questions about predicting expl performances in rock from performance in water

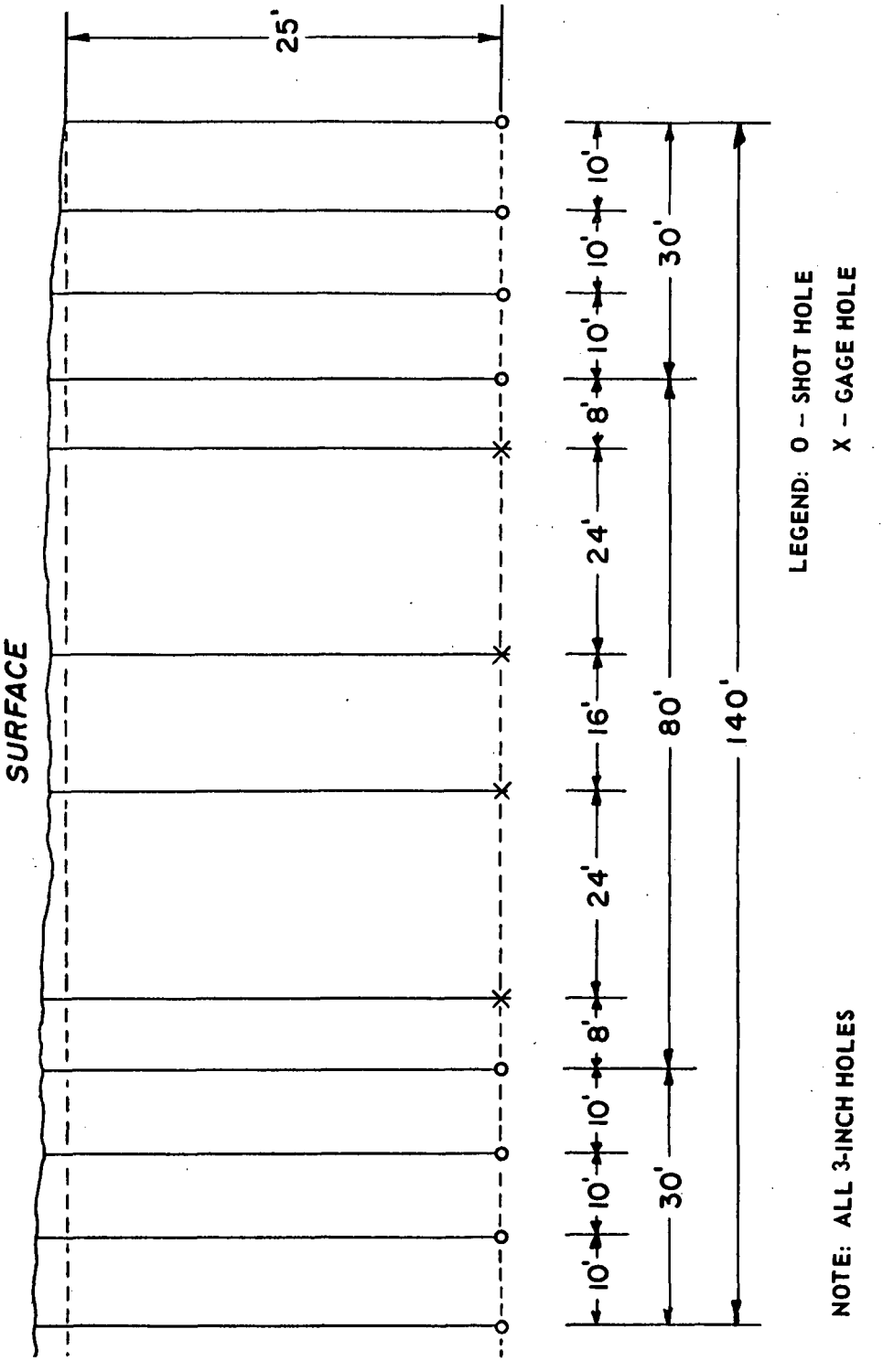
The purpose of the tests described on the paper of Missouri School of Mines (Ref 7) is to help in predicting expl performance in rock from perfomance in water. Four expls were tested both in water and in rock so that direct comparison of the results of the two methods of testing could be made. The rock tests were made at a granite quarry operated by the Consolidated Quarries Division of the Georgia Marble Co, near Lithonia, Ga. The rock was uniform with sp gr ca 2.6 and propagation velocity of about 18000 ft/sec. The underwater tests were made in a deep pond at the Armour Foundation test facility near Coal City, Illinois. Water had sp gr 1.0 and propagation velocity 5000 ft/sec

The expls tested were a) **HP-G**-Hercules 60% High Pressure Gelatin (initiated by No 6 Blasting Cap); b) **N-IV**-Spencer N-IV AN-FO expl consisting of 94/6-AN prill-Fuel Oil mixture with a small amt of surface-active agent to prevent caking (initiated to high-rate underwater detonation by No 6 cap and 36-inch long 400-grain Primacord); c) **MN-H**-Spencer Mr "N" AN-FO expl consisting of 94/6-AN prill/Fuel Oil with 3% diatomaceous earth added as coating to prevent caking (initiated as N-IV expl to high-rate detonation) d) **MN-L**-Spencer Mr "N" AN-FO. Same as MN-H but initiated with No 6 cap and 12-inch of 400-grain Primacord in order not to produce high-order detonations. Eight pounds of expl chges were used in all of the tests except some of the High Pressure Gelatin shots in the underwater tests were 1.3 pounds

The strain measurement test set-up consisted of linear array of shot and gage holes, as shown in Fig Ex 15. The holes were drilled in solid rock about 25 ft deep and at a distance from any free face, so that charges detonated at the bottom of the shot holes did not produce surface breakage, and so the gages cemented in the bottom of the gage holes recorded the strain pulses produced in the rock without interference from surface reflections. Four holes at each end of the array were used as gage holes. Thus two shots with each expl were made in essentially the same rock and using the same gages. This provided sufficient information for a statistical analysis of the results

Some details of the shot and gage holes are shown in Fig Ex 16. Eight pound chges loaded into 3-inch holes resulted in charge-lengths of about 2 ft for the Hercules HP-G chge and about 3 ft for Spencer AN-FO chges Before being loaded each chge was weighed and divided into several small increments. A No 6 Blasting Electric Cap was taped to a piece of ¼-inch dowel, having the same length as the chge. For the AN-FO chges the Primacord booster was cut in short lengths and taped around the cap. To measure the rate of deton two targets were taped to the dowel, are 1st one about 3 inches above the cap and the other near thetop of the dowel. A microsecond chronograph recorded the difference in arrival time of the deton wave at the two targets. The dowel was lowered to the bottom of the shot hole and held upright at one side. The HP-G chges were loaded by slitting 1¼ by 8 inch sticks and tamping small increments in the hole. The AN-FO chges were loaded by pouring in small increments. Both types of expls completely filled the space at the bottom of drill hole and on top of chges sand was placed to stem the shot. Rock breakage was limited to a crushed zone see Fig Ex 16 in the immediate vicinity of the chge. After each shot the crushed zone was cleaned by blowing the hole with compressed air. Then the volume of the crushed area was measured by adding sand in known volume increments. The order of shooting was from the outermost shot hole in towards the gage holes, alternating from one end of the array to the other, so that for no shot was there any already broken rock betn the chge portion and the gages

The gage used to record the strain pulse produced in the rock was a radial strain gage consisting of a resistance-wire sensing element shielded with Cu and bonded to a short



LINEAR ARRAY OF TEST HOLES

FIG Ex15

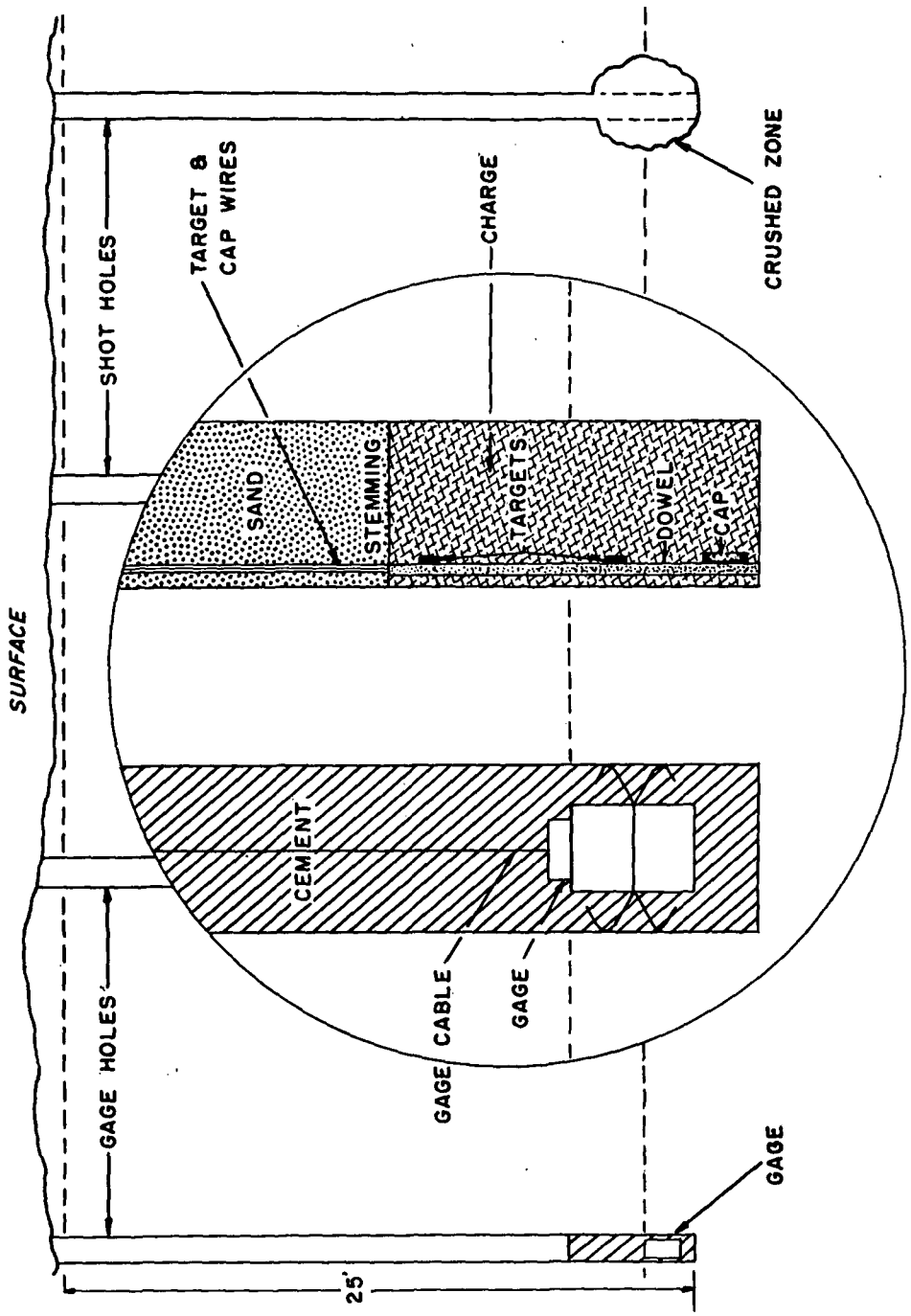


FIG Ex16

DETAIL OF LINEAR ARRAY TEST

length of rock core. It was placed near the bottom of the gage hole, oriented to measure horizontal strain in the direction of the array of holes, and fixed in place with a high-strength cement. Cables led from the gages a distance of about 500 ft to recording equipment housed on an instrumental trailer. On each shot a recording was made from each of the four gages providing a strain-amplitude versus time plot of the radial strain at each gage position. From the records it was possible to determine the arrival time, amplitude and other characteristics of the strain pulse produced by each shot as a function of the travel distance of the pulse

The underwater measurements were made using the following technique: expl chges were detonated at a depth of ca 25 ft in water ca 45 ft deep. Piezoelectric pressure gages were located at the 25 ft depth at distance of 25 and 50 ft from the chge (Fig. Ex 17). The location of the chges and the gages were chosen in the manner not to have reflections from the surface or the bottom interfere with the direct pressure pulse. The chges were prepd by loading the expl, primed with No 6 Electric Blasting Cap and Primacord Booster if required, into waterproof plastic bags. Each chge was weighted with a bag of sand hung below the chge, and then lowered on a line to a desired position. Gage cables and fire line led about 500 f t to recording instruments located in a trailer. On each shot a pressure versus time recording was obtd from each gage and each record made possible the determination of the amplitude, & other characteristics, and of the initial pressure pulse produced by the shot. By measuring the interval betn the primary and secondary pressure pulses, it was possible to determine the period of the oscillating gas bubble produced by the shot. The energy associated with the gas bubble was then calcd from the period of oscillation

More detailed description of experimentation is given in Ref 7 on which also are included several tables and charts. We are

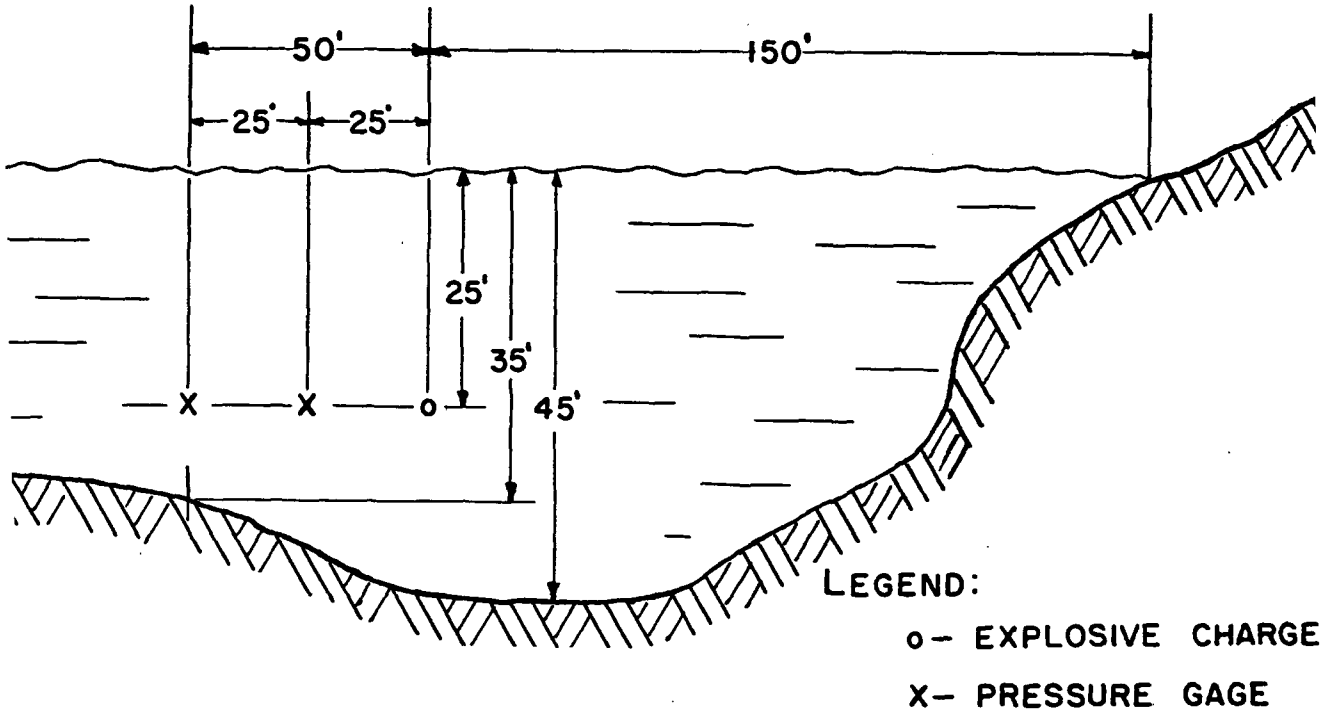


FIG Ex17

UNDERWATER TEST SETUP

giving here a summary of results which are in Table 4 of Ref 7, reproduced here as Table Ex7. This table shows relative values of the performance parameters for the four expls based on values for HP-G equal to 100. In comparing the results of the two test methods a reasonable criterion for determing significant differences among the parameters is a separation of at least two standard errors. With this in mind the following conclusions can be drawn: a) The various performance parameters rank the expls in the same order of expected effectivness, showing general agreement betn the rock and water test methods b)The underwater tests show a significantly greater difference betn MN-H and MN-L than do the rock tests. The addnl confinement provided by the rock evidently caused the MN-L to detonate at or near its high rate, even though initiated with a small booster which produced a low rate of deton in the water tests. This was confirmed by the nearly equal deton rates measured in the rock tests for the MN-H & MN-L c) The peak strain oroduced in the rock showed a signific

Table Ex7
Relative Performance Parameters

Rock Tests

	Peak strain	Strain impulse	Strain energy	Crushed volume
HP-G	100	100	100	100
N-IV AN-FO	70	110	81	68
MN-H	48	85	40	65
MN-L	41	69	26	37
Average standard error (%)	15	18	30	15

Water Tests

	Peak pressure	Pressure Impulse	Pressure energy	Bubble energy
HP-G	100	100	100	100
N-IV AN-FO	102	89	99	80
MN-H	86	76	56	77
MN-L	42	49	25	31
Average standard error (%)	10	8	15	10

cantly greater difference betn the HP-G and the two fully-boostered AN-FO expls than did the peak pressure produced in the water. This difference may be explained by the different impedance ratios involved in the transfer of pressure from expl to rock and from expl to water. The differences in confinement provided by rock and water, and the differences in expl-rock and expl-water impedance ratios, must be kept in mind when predicting expl performance in rock from performance in water

Refs: 1) W.I.Duvall & T.C.Atchison, "Rock Breakage by Explosives", USBurMines Rept of Investigation **RI5356** (1956) 2) W.I.Duvall & B. Petkof, "Spherical Propagation of Explosion-Generated Strain Pulses in Rock" USBurMines **RI 5483** (1959) 3) T.C.Atchison & W.E.Tournay, "Comparative studies of Explosives in Granite", US Bur Mines **RI 5509** (1959) 4) D.E.Fogelson et al, "Strain Energy in Explosion-Generated Strain Pulses", US BurMines, **RI5514**(1959) 5) T.C. Atchison& J. Roth, "Comparative Studies of Explosives in Marble", USBurMines **RI5797** (1961) 6) L.D.Sadwin et al, "Underwater Evaluation of the Performance of Explosives", International Symposium on Mining Research Rolla, Missouri, Feb 1961 7) T.C. Atchison, S.J. Porter & W.I. Duvall, "Comparison of Two Methods for Evaluating Explosive Performance", Intnl Symp Mining Research, Rolla, Mo, Feb 1961

Explosive Phenomena and Detonation. See Detonation and Explosives Phenomena in Vol 4, p D346-R

Explosive Picratol. See Explosive Composition Picratol

Explosive Pipe. See under Explosive Rivets

Explosive Pipe Closure System. A fast, high-strength mechanism for closing off inaccessible pipes is explosively actuated. The system, devised to protect instrumentation mounted in pipes from flying debris during uunderground nu-

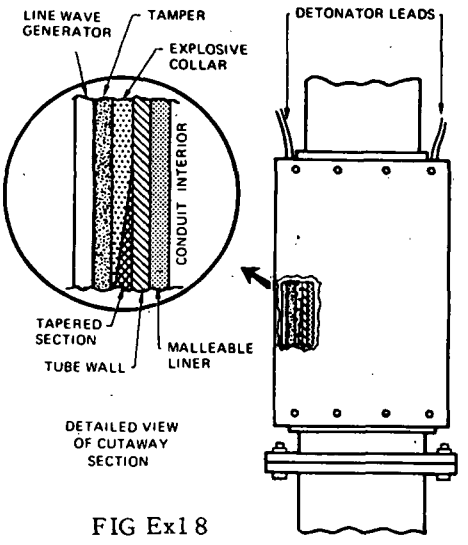


FIG Ex18

clear testing can also be used to close oil well casings or other piper in hard-to-reach locations. A 4-inch ID pipe was closed in 200 microseconds. By comparison, conventional mechanical valve closures require a longer actuating time, particularly as the pipe diameter increases

The design consists basically of a tubular steel section of pipe with a malleable metal liner on the inside and an expl assembly on the outside (Fig Ex 18), Detonators initiated at opposite sides of the expl initiate a detonation wave along two line-wave generators to a cylindrical expl collar located beneath a lead tamper. The expl then generates a radial shock wave that causes the pipe and liner to implode and close. The pipe wall is thicker at the point where the cylindrical expl charge begins, tapering down to the minimum pipe diameter. The expl cross-section also thickens as it approaches the thinner portion of the pipe wall. These two features prevent the liner from breaking up before a complete closure is achieved. The design specifies that the length of the explosive collar should be about 2.5 to 3 times the diameter of the pipe aperture, and that the pipe taper length should be 1.5 times this diameter. For every square inch, the expl content of the cylindrical charge should range from about 4 to 12 grams per square inch per inch of wall thickness

Refs: 1) Sandia Laboratories, Albuquerque, New Mexico, 87115 2) G.Cohn, Edit, Expls & Pyrots **5** (5), 1972

Explosive, Plastic. See under individual names

Explosive PLX. See Explosive Composition PLX

Explosive Power. See under Explosive Properties and as Power of Explosive in Vol 1 of Encycl, p XX

Explosive Power vs Oxygen Balance, Correlation of. The relationship of oxygen balance to expl power as measured by ballistic mortar has been studied empirically. Starting from modified oxygen, balance developed for detonation velocity calculations, a numerical measure called the "power index" is derived which correlates closely the features of molecular structure with the power values. Expl power has also been expressed as an additive function of details of molecular structure. This is the basis for a method whereby power values may be derived which agree with experimental results to about 5%

Ref: A.R.Martin & H.J.Yallop, JApply Chem **9**, 310-15 (1959) & CA **53**, 19389 (1959)

Explosive Press. An explosively activated device by which materials may be compressed at pressures estimated at several millions psi. The presses may comprese either single or double-acting pistons. In a press consisting of a single-acting piston (Fig Ex 19), the sample is placed in the cavity of the lower platen. The steel piston is inserted on top of the sample and the upper platen is placed in position. A cylinder of high expl (Comp B) is placed at the center of the upper platen and detonated by a standard detonation. The piston is driven into the lower platen compressing the sample

For higher pressures, double-acting pistons are employed (Fig Ex 20). In this press, the center plate has a lapped hole in which 2 pistons move. The sample is placed between the

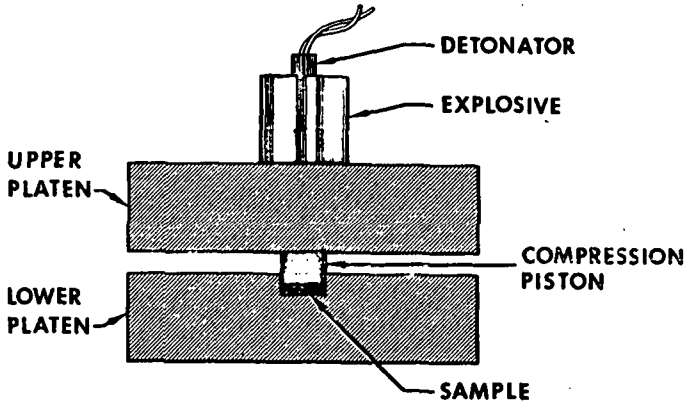


FIG Ex19. CROSS-SECTIONAL VIEW OF A SINGLE-PISTON PRESS

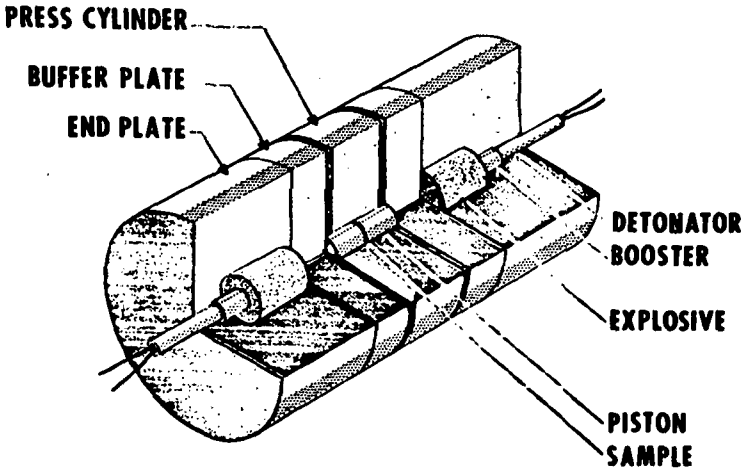


FIG Ex20. CROSS-SECTIONAL VIEW OF A DOUBLE-PISTON PRESS

2 pistons. Buffer plates are inserted between the pistons and expl charges. These buffer plates drive the pistons when the expl charges in the steel end plates are simultaneously activated

Ref: E.W.La Rocca & J. Pearson, "The Explosive Press as a Research Tool in Material Behavior and Forming" NOTS Rept **2019** (1958), NAVORD rept **5880** (1958)

Explosive Product Laser. The principle of using the products & energy from an expl as the medium for a laser pulse was studied by Hershkowitz & Lanzerotti (Ref 2) who also designed a laser detonation cartridge (LDC). The LDC uses the products from the deton of an expl whose composition, shape and mode of initiation were optimized. Interaction with a deformable wall directs the products thru convergent-divergent nozzles producing an inversion suitable for lasing

Expls were chosen to approximate the known molecular requirements of a CO_2 laser. Product compn, temp & press were calcd for an isentropic expansion from the Chapman-Jouguet detonation point (See Vol4 of Encycl,pD231-R). Effective upper & lower CO laser level relaxation times for the products were calcd and compared with flow expansion times to determine if inversion conditions were fulfilled. Results are presented for PETN, TNMe &

HNB, alone and mixed with Al. Predictions were verified by subsequent comparison with available exptl data

The expansion of products from an axisymmetric expl was calcd using the HEMP computer program (See Vol 4, pD180 and Refs 12 & 15 under COMPUTERS), which gave data for the calcn of further flow, and of the interaction of the products with the wall using the STROBE program. STROBE is a modification of a program written during investigation of muzzle blast (Ref 1)

It was predicted that an LDC with 30 g of expl in a 175 mm cartridge of 6 mm wall thickness and with an inner chamber of 120 mm max diam will provide 60 kilojoules total energy yield over a one millisecond duration in a pulsed flow mode

Note: Lasers are briefly described under "Detonation, MASER AND LASER in" in Vol 4, pp D436-L to D441-L

Refs: 1)T.D.Taylor, "Calculation of Muzzle Fields", PATR 4155 (Dec 1970) 2)J.Hershkowitz & M.Y. DeWolf Lanzerotti, "Preliminary Design Study for an Explosive Product Laser", PATR 4465, (April 1973), 39 pp with 28 refs

EXPLOSIVE AND RELATED PROPERTIES INCLUDING TESTS, OF EXPLOSIVES AND PROPELLANTS

1. Ability to Propagate Detonation or Transmission of Detonation. See Vol 1 of Encycl, pVII

2. Armor Plate Impact Sensitivity. See Vol 1, pVII and AMCP 706-177 (1967), p 7

3. Ballistic Mortar and Ballistic Pendulum Tests are listed under Power or Strength and briefly described in Vol 1, pp VII & VIII

4. Behavior Towards Flame and Heat. See under Sensitivity to Flame, Heat, Sparks, Electrostatic Discharge

5. Bichel Bomb Pressure Gage. See Vol 1, p VIII and in Vol 2, under Closed Bomb, pp C330 to C331

6. Blast Effect (BLE). It is the ability of an expl charge to destroy matter, or structures, located far from the site of expln

More detailed discussion is given in Vol 2, of Encycl under BLAST EFFECTS IN AIR, EARTH AND WATER, pp B180-L to B-184-R. Also in Vol 4, p D149-L, under Blast,Spherical

Accdg to the late W.R.Tomlinson Jr. formerly of Picatinny Arsenal, blast effect is measured by the distance from burst, to which a certain type of damage occurs. Four types are distinguished a)Confined air blast; b) Free air blast; c)Underground blast; and d) Underwater blast

Blast action is such that, for an oxygen balanced expl, the heat of expln appears to be propagated thru the medium (such as air) in the form of a shock wave, or at least BLE-He (heat of explosion). For a negatively balanced compn, or one in which after-reactions (usually exothermic) can occur, BLE-He + A where A is the heat of the after-reaction. In air, the after-reaction of most importance is the air-oxidation of unoxidized portions of the charge especially of the exothermic elements like Al. When the same elements are oxidized under water by CO_2 or H_2O, it is part of He

Blast effects can be tested by various methods such as listed in Vol 2, pp B214-R & B215-L, under Blast Meters

Blast Effectiveness of Various Explosives is given in Table on p B182 of Vol 2

Blast Effects Due to Reflected Shock Waves is given in Vol 2, p B182-L

Blasting Action of High Explosives in Relation to Their Explosive Properties is discussed by Cook (1958), Chap 11, pp 265-82 (9 refs)

Blast Contours is discussed by Cook (1958), pp 106-14 and listed in Vol 4 of Encycl (1969), p D148-L

Blast Potential is discussed under Damage Potential of Air and Ground Blast Waves in Vol 3, p D3-R

7. Booster Sensitiveness. It is the relative sensitiveness to an arbitrary graded series of boosters. One of its tests is briefly described in Vol 1, p8. As booster sensitivity is considered as one of the "Detona-

tion by Influence or Sympathetic Detonation", described in Vol 4, pp 395-R to D398-R, it can be tested by the following method:
Barrier Test (Vol 4, p D331 & Fig 16)
Card-Gap Sensitivity Test (Vol 4, p D398-R)
Card Test (Vol 4, p D339-L)
Coefficient de self-excitation (CSE)-Fr test described in Vol 3, p C390-L
Cylindrical, Gap Test (Vol 4, p D333-L & Fig 19)
Four Cartridge Test (Vol 4, p 399-L)
Gap Tests (Vol 1, p XIV and in Vol 4, pp D303-R to D304-L)
Halved Cartridge Gap Method (Vol 1, p XIV
Large Scale Gap Test (LSGT) (Vol 4, p D318-R & Fig 2 on p D319-L. Also p D324)
NOL (Naval Ordnance Laboratory) Gap Test (Vol 4, p 321-L & Fig 4)
NOL Modified Gap Test (Vol 4, p 327 & Fig 8 and pp D335-R & D336-L with Fig 26
Perpendicular Gap Test (Vol 4, p 332-R & Fig 18)
Shock-Pass-Heat-Filter (SPHF) Sensitivity Test (Vbl 4, p D316 with Fig 1 and p D399-1)
Small Scale Gap Test (SSGT) (Vol 4, p D343-R & D344 with Fig 35)
Three-Legged Table Sensitivity Test (Vol 4, p D399-R)
Wax Gap Test (Vol 1, p VIII, under Booster Sensitivity and p A354, Note e, under Ammonium Nitrate. Also Vol 4, p D329 with Fig 13)
Whole Cartridge Sensitivity Test (Vol 4, p D399-R
[See also AMCP **706-177** (1971), p 6]

8. BRISANCE (B). It is the ability of an expl charge to deform, fragment, or shatter matter at, or close to its surface

A detailed discussion is given in Vol 2, under BRISANCE or SHATTERING EFFECT, pp B265-L to B266-R. Table 1 listed on pp B266 to B298 gives for numerous expls brisance tests values in relation to TNT taken as 100. These tests include **CCCT**-Copper cylinder compression test; **FET**-Fragmentation efficiency test; **FGT**-Fragment gun test; Kast's Formula values; **LBCT**-Lead block compression test; **PCT**-Plate cutting test; **PDT**-Plate denting test and **ST**-Sand test. References for these and some other brisance tests are given under "Brisance Test Methods" on p B299.

Two Ger methods: "Strahlungsprobe" (Radiation Test) and "Stanzprobe" (Punch Test) are described on pB299

The following brisance tests are described in Vol 1 of Encycl:
Fragmentation Tests (p XII)
Fragment Gun Test (p XII)
Hopkinson Pressure Bar Test (p XVI)
Plate Denting Tests (p XIX)
Plate Tests (p XX)
Quinan Test (p XXI)
Sand Test (pp XXI & XXII & PATR **3278** (1965), p 16
Modified Sand Test to Determine the Brisance of Liquid Explosives is described in PATR **3278** (1965), p 16

The following brisance tests are described in Vol 3 of Encycl:
Closed Pit (or Chamber) Test and Other Fragmentation Tests, which include:
Closed Pit Test (p C346-L & Fig on p C347)
OpenPit Test (p C346-R & C349-L)
Panel Test (p C349 & Fig)
Fragment Velocity Test (p C350-L)
Low Panel Test (p C350-R)
Silhouette Test (p C350-R)
High Panel Test (p C350-R)
Splitter dichteprobe (Fragment Density Test), (p C350-R)
Compression (or Crusher) Tests, which include:
LBCT (Lead Block Compreesion Test), called in Ger Stauchprobe nach Hess (Hess' Crusher Test), uses apparatus "Brisance Meter of Hess" (p C492 with Fig). Its French version is known as Épreuve des petits plombs (Test with Small Leads) (pp C492-R & C493-L). Another Fr modification is Epreuve de Chalon (p C493-L). In the US modification of Hess's method only one lead block is used. It is called Compression Test with Small Block (pC493-L).(Copper Cylinder Compression Test), called CCCT in Ger Stauchprobe nach Kast (Kast's Crusher Test), uses apparatus Brisance Meter of Kast (p C493-with Fig)
Compression Test of Quinan permits detn of not only brisance, but also of strength (potential) (pp C493-R & C494-L with Fig)

The following brisance subjects are discussed in Vol 4:

Brisance or Shattering Effect (pp D149-R & D150-L)
Brisance, Correlation with Chemical Structure (p D150-L&R)
Brisance, Correlation with Properties Other Than Chemical (p D150-R)
Brisance Determination by Method of Metal Accelaration by Explosives (pp D150-R to D152-R)

8a. Brisance-Detonation Velocity Relationship is discussed in Vol **2**, p B297. Table on p B298 gives for numerous expls, Brisance Density and Detonation Velocities calcd by the late W.H.Rinkenbach and those taken from the literature

9. Bulk Compressibility and Bulk Modulas. See Vol **1**, p IX and AMCP **706-177** (1967), p 9

10. *Bullet (or Rifle Bullet) Impact Sensitiveness.* See Vol **1**, p IX and under impact (or Shock) Sensitiveness. Also "Rifle Bullet, Impact Test" in AMCP **706-177** (1971), p 2

11. Burning or Combustion. See Vol **2**, p B343 and Vol **3**, p C425 & C426

12. Burning (Combustion) and Burning (Combustion) Characteristics of Explosives, Propellants and Pyrotechnic Compositions. See Vol **2**, pp B343-R to B356-R. Also Vol **3**, pp C427-L to C430-L

13. Burning Erosive in Propellants. See Vol **2**, p B357

14. Burning Rate, and Burning Rate Coefficient. See Vol **2**, p B359-R

15. Burning Rate of Propellants as a Function of Pressure. See Vol **3**, p C335 with Fig of pressure vessel

16. *Burning (or Combustion) Rate Test and Burning Test* are described by Ch.E. Munroe & J.E. Tiffany on pp 30-31 of "Physical Testing of Explosives", USBurMines Bulletin **346**, Govt Prtg Off, Washington, DC (1931)

17. Calorimetric Tests. See Heat of Formation, Combustion, Deflagration, Explosion and Detonation

18. Capability to Inflame Test. French test known as Épreuve de l'aptitude de l'inflammation, described by L. Médard, MP **33**, 329-30 (1951)

19. Cavity Charge Efficiency. See Shaped Charge Efficiency

20. Characteristic Product of Berthelot or Force spécifique, in Fr. See Berthelot's Characteristic Product in Vol **2**, pp B105 & B106-L

21. Chronographs and Other Devices Used for Determination of Detonation Velocities. See Vol **3**, pp C310-R to C319-R

22. Closed Bomb (or Vessel) and Instruments for Measuring Pressures Developed by Explosives and Propellants. See Vol **3**, pp C330-L to C345-R

23. Closed Pit (or Chamber) Test and Other Fragmentation Tests. See Vol **3**, pp C345-R to C351-L

24. Coefficient d'utilisation pratique (CUP). (Épreuve de travail spécifique). French test for detn of power or strength. It is modification of Trauzl Lead Block Test. See Vol **1**, pp IX & X

25. Combustion, Combustion Rates and Combustion Tests. See Burning or Combustion (item 11) and items 12, 14, 15 & 16

26. Compression (or Crusher) Tests. See Vol **3**, pp C492 to C494

27. Concrete Test. See Vol **3**, p C495-L

28. Cook-off Test of Spencer Chemical Co. See Vol **1**, p A354, Note a

29. Covolume. See Vol **4**, p D196-L

30. *Cratering Effect Tests.* See Vol 3, pp C554 to C555-L

31. *Crawshaw-Jones Apparatus for Testing Coal Mine Explosives for Permissibility.* See Vol 3, p C555-R

32. *Crusher Tests.* See Compression (or Crusher) Tests

33. *CUP or cup.* See Coefficient d'utilisation pratique

34. *Dautriche Method.* See Vol 3, p C311-R

35. *Decomposition Temperature Test* is described by A.J.Clear in PATR 3278 (1965), p 8

36. *Deflagration Temperature Test.* See Detonation (Explosion, Deflagration or Ignition) Temperature Test

37. *Density Determinations* are described in Vol 3, pp D64-R to D81-R

38. *Detonability and Flammability of Explosives, Propellants and Pyrotechnic Compositions* is discussed in Vol 4, pp D211-R to D213-R

39. *Detonating Capacity of Explosives; Effect of Various Factors* is discussed in Vol 4, pp D213-R to D216-R

40. *Detonation (or Explosion) by Influence or Sympathetic Detonation.* Its definition and description are given in Vol 1, p X and in Vol 4, pp D395-R to D398-R. Its tests are listed on pp D398-R to D402-L and also under Booster Sensitivity Tests.

41. *Detonation (or Explosion) Pressure.* Its definition is given in Vol 1, p X and description in Vol 4, pp D483-L to D485-R. It must not be confused with Pressure of Gases Developed on Detonation defined in Vol 1, pXX

Experimental determinations of pressures developed on detonation or expln are listed and in some cases, briefly described in Vol 4, D485-R to D491-R. More complete descriptions of some of these tests are given under "Closed Bomb (or Vessel) and Instruments for Measuring Pressures Developed by Explosives and Propellants", in Vol 3, pp C330-L to C345-R

42. *Detonation Temperature.* See Explosion (Deflagration or Ignition) Temperature. Not to be confused with Temperature Developed on Detonation or Explosion

43. *DETONATION VELOCITY(Rate of Detonation or Velocity of Detonation) AND TESTS.* Its definition and list of 14 refs are given in Vol 1, p X. A detailed discussion on this subject is given in Vol 4, under the following items:
Rate of Detonation or Velocity of Detonation (pp D629-R to D640-R with 85 refs
Detonation Velocity, Anomalous High (pp D640-R to D641-L)
Detonation Velocity-Charge Diameter Relationship (pp D641-L to D643-L)
Detonation Velocity-Charge Density Relationship (pp D643-L to D646-L)
Detonation Velocity - Charge Diameter and Density Relationship (pp D646-L to D654-R)
Detonation Velocity and Chemical Composition and Detonation Velocity as a Function of Oxygen Balance and Heat of Formation (pp D656-L to D657-L)
Detonation Velocity-Confinement and Obturation Relationship (pp D657-L to D660-L)
Detonation Velocity - Critical and Limiting Diameter Relationship (p D660-L & R)
Detonation Velocity by Direct Visualization of the Explosive Flow (pp D660-R to D662-R)
Detonation Velocity, Effect of Replacement of Air in Explosive Charges by Non-Explosive Liquids (p D663-L)
Detonation Velocity in Gases (pp D663-L to D666-L)
Detonation Velocity, Influence of Ageing in Gelatin Explosives (pp D666-L to D667-L)
Detonation Velocity, Influence of Inert Components and Inert Additives (pp D667-R to D668-R)
Detonation Velocity, Influence of Magnetic, Electro-Magnetic and Electrical Fields as well as of Electrons On (pp D668-R to D671-L)

Detonation Velocity, Influence of Method of Initiation (pp D671-L to D672-L)
Detonation Velocity by Metallic Transmission of Sulfur (p D672-L & R)
Detonation Velocity - Particle Size Distribution Relationship (pp D672-R to D673-R)
Detonation Velocity - Pressure Over Explosive Relationship (pp D673-R to D674-R)
Detonation Velocity - Temperature (Initial) of Charge Relationship (pp D674-R to D675-R)
Detonation Velocity, Experimental Procedures are listed in Vol 4, pp D654-R to D656-R and described in Vol 3, pp C310-R to C319-L under CHRONOGRAPHS. The following methods are included:

Dautriche Method (pp C311-R to C312-R)
Mettegang Chronograph Method (pp C313-L and Fig on p C312)
Pin Chronograph Method (pp C313-R to C315-R)
Chronographic Method Employing Microwave Technique (pp C315-R)
Cook's Method, Pinset Diagram (p 316)

43a. Detonation Velocity - Brisance Relationship. See Item 8a: "Brisance - Detonation Velocity Relationship"

44. Explosion (Detonation, Deflagration or Ignition) Temperature. It is the temperature at which an explosive, propellant or pyrotechnic composition explodes, detonates, deflagrates or ignites within a definite time, say 0.1, 1.0 or 5.0 seconds. Various methods for its determination arebriefly described in Vol 1, p XVI under Ignition (or Explosion) Temperature Tests

A detailed description of the test conducted at Picatinny Arsenal is given by A.J. Clear in PATR **3278** (1965), pp 7-8 with Fig. 8 on p 39

See Also Vol 4, pp D390-R to D391-R and D583-L to D589-L

45. Exudation (or Sweating) of Explosives. See Vol 1, p XI with 7 refs

46. FI (Figure of Insensitiveness) Test. See Vol 1, p XII

47. Flame Test. See Vol 1, p XII

47a. Flammability Index. See AMCP **706-177** (1967), p 3

48. Flash Point. The lowest temperature at which a combustible or explosive liquid will give off a flammable vapor which will burn momentarily. It can be conducted in a closed or or open cup (See under F's)
Ref: CondChemDict (1961), 499-R

49. Fragmentation Tests. See under BRISANCE and under "Closed Pit (or Chamber) Test and Other Fragmentation Tests" in Vol 3, pp C345-R to C351-L

50. Friction Sensitivity. It is a measure of the resistance of an expl to friction, one of the properties required for determining safety in handling and transportation. Several methods for its determination are briefly described in Vol 1, p XIII

51. Gap Tests. See Vol 1, p XIV and Vol 4, pp D303-R to D304-L

52. Gas Pressure Developed on Explosion or Detonation is defined under Pressure of Gases Developed on Explosion or Detonation in Vol 1, p XX. See Also under Detonation (and Explosion) in Gases in Vol 4, p D352

53. Gas Volume Developed on Explosion or Detonation. See Volume of Gases Evolved on Explosion or Detonation in Vol 1, p XXVI

54. Heats of Formation, Combustion, Deflagration, Explosion and Detonation. See Vol 4, Section 5, pp D369-L to D384-R

55. Heat Tests. See Vol 1, p XV and under Stability and Instability of Explosives

56. Heaving Action. See under Propulsive Action

57. Hydrolysis Tests. See AMCP **706-177** (1971), p 9

58. Hygroscopicity. See Vol 1, p XVI & AMCP **706-177** (1971), p 3

59. *Ignition Temperature.* See Vol 1, p XVI and under Explosion (Detonation, Deflagration or Ignition) Temperature

60. *Impact (or Shock) Sensitiveness.* It is a measure of the resistance of expls to impact by a definite weight dropping from various heights. The test, known as Drop (or Falling Weight) Test serves to determine safety in handling and transportation. Its brief description is given in Vol 1, p XVII and in Vol 4, pp D391-R to D392. Another impact test, known as Bullet (or Rifle Bullet) Test is described in Vol 2, pp B332-L to B334-L with Tables on pp B335 to B339
Note: A.J.Clear gives in PATR 3278 (1965) detailed descriptions of "Impact Test with Picatinny Arsenal Apparatus" (pp 2-4 with Fig 4 on p 35) and "Impact Test with US Bureau of Mines Apparatus" (pp 4-7 with Fig 7 on p 38). He also describes on p 7 "Modified Impact Tests for Liquid Explosives", made with US Bureau Mines and PicArsn Apparatuses

61. *Impetus and Available Energy in Detonation.* See Vol 4, pp D391-R & D393-L

62. *Impulse in Detonation.* See Vol 4, pp D393-L to D394-L

63. *Impulse Specific in Detonation.* See Vol 4, p D394

64. *Index of Enflammability .* See Vol 1, p XVII and AMCP 706-177 (1971), p 3

65. *Induction Period in Initiation of Explosives, Propellants and Pyrotechnic Compositions.* See Vol 4, pp D394-R to D395-L

66. *Initiating Efficiency.* See Vol 1, p XVIII

67. *Initiation Sensitivity .* See Sensitivity to Initiation

68. *Munroe-Neumann Effect Efficiency.* See Shaped Charge Efficiency

69. *Percussive Force of Explosives.* See Vol 1, p XIX

70. *Permissibility of Coal Mining Explosives* is determined by submitting them to tests described in Vol 3, pp C368-R to C378-L. The galleries used for testing are briefly described in Vol 1, p XIV and more fully in Vol 3, pp C370-R to C378-R

71. *Plate Tests.* See Vol 1, p XX and under BRISANCE

72. *Potential* (Fr Effet utile). See Vol 1, p XX

73. *Power or Strength.* It is a measure of the capacity of doing useful work . It refers to both blast and propulsion, but *power* is normally used in military parlance, while *strength* by the commercial expl industry. See Vol 1, p XX and in Vol 4, pp D476-L to D479-R

Power or strength is usually detd by one or several of the following tests:
Ballistic Mortar (Vol 1, p VII)
Ballistic Pendulum (Vol 1, VII & VIII)
Cratering Effect (Vol 3, p C554)
Quinan Apparatus (Vol 1, p XXI & Vol 3, pp C493-R & C494-L)
Trauzl or Lead Block Expansion (Vol 1, p XXV)
Mortar (Vol 1, p XIX)

74. *Pressure of Detonation (or Explosion).* See Detonation (or Explosion) Pressure

75. *Pressure of Gases Developed on Explosion or Detonation.* See Vol 1, p XX

76. *Propagation of Explosion or Detonation.* See Ability to Propagate Detonation or Transmission of Detonation in Vol 1, p VII

77. *Propulsive Action (PrA) or Heaving Action (Propulsive Force).* It was defined by the late W.R. Tomlinson Jr, formerly of Picatinny Arsenal, as the ability of an expl or propellant charge to propel, or hurl matter or a projectile. This term has been used in industry to rate commercial expls and represents the same property as ballistic potential defines for propellants. PrA is proportional to the "hot-gas-volume" PrA = nRT, where n = gas volume per gram of the expl product at STP, R = gas cons-

tant and T = absolute temperature. Tests which can be used:
Ballistic Mortar (Vol 1, p VII)
Ballistic Pendulum (Vol 1, p VII)
Mortar (Vol 1, p XIX) [Barnett (1919), 181-82]
Note: Accdg to L.V.Clark, IEC **25**, 1388 (1933), the *relative propulsive strength* can be determined either by Ballistic Pendulum Test or Trauzl Test, usual tr sts for power determination. The Pb block compression value is regarded as a measure of relative brisance while Fragmentation tests in hand-grenade bodies measure relative shattering power

78. *Quickness (Vivacité in Fr) of Burning of Propellants Determination.* See H.Muraour ''Poudres et Explosifs'', Paris (1947), 90-92 and also his papers published in MP and MAF

79. *Resistance to Heat Tests* (Épreuves de la résistance à la chaleur). French ''official'' described in book listed in Vol 1, p XXI

80. *Rifle Bullet Test.* See Bullet Impact Sensitiveness Test

81. *Sand Test and Sand Crusking Test.* See Vol 1, p XXI & XXII and under BRISANCE

82. *Sensitivity to Detonation by Initiating Agents.* See Sensitivity to Initiation

83. *Sensitivity to Electrostatic Discharges.* See "Electrostatic Sensitivity Test" in PATR **3278** (1965), pp 14-15 and Fig 13 on p 44 and also "Sensitivity to Initiation by Electrostatic Discharge" in AMCP **706-177** (1971), pp 9-10

84. *Sensitivity to Flame, Heat and Sparks.* See Vol 1, pp XXII & XXIII

85. *Sensitivity to Impact.* See Impact Sensitivity

86. *Sensitivity to Initiation by Primary Explosives, Detonators and Boosters.* It can be expressed as the minumum weight of an initiating expl or the No of detonators to cause a complete detonation of a HE (high expl). See Vol 1, p XXIII and under Detonation, Sensitivity of Explosives to, in Vol 4, pp D511-R to D515-R. See also A.J.Clear, PATR **3278** (1965), pp 9-11: "Sensitivity to Initiation as Determined by Sand Test" and on pp 12-14: "Sensitivity to Initiation by the Modified Sand Test for Liquid Explosives"

87. *Shaped Charge Efficiency.* See Vol 1, pp XXIII and under Detonation, Munroe-Neumann Effect in Vol 4, pp D442-R to D454-L

88. *Stability and Instability of Explosives* are discussed in Vol 4, pp D569-L to D574-L. The so-called *Thermal Stability* can be detd by the following Heat Tests:
Abels or KI-Starch Test (Vol 1, p A2-L)
Angeli's Test (Vol 1, p A403)
Bergmann-Junk Test (Vol 2, pp B102-R & B103-L.)
Brame's Test (Vol 2, p B260)
Brunswig's Test (Vol 2, p B319-L)
Chiaraviglio & Corbino's Test (Vol 2, p C179-L)
Desmaroux's Test (Vol 3, p D90-R)
German 132° Test (Vol 1, p XV)
Heat Tests at 100°, 120° & 134.5° (Vol 1, pp XV & XVI)
International Test at 75° (Vol 1, p XVIII)
Resistance to Heat Test (Vol 1, p XXI)
Silvered Vessel Test (Vol 1, p XXIV)
Surveillance Tests at 65° & 80° (Vol 1, p XXIV)
Taliani's Test (Vol 1, pp XXIV & XXV)
Vacuum Stability Tests (Vol 1, p XXVI)

Several tests which are listed in Vol 1, p XV under Heat Tests (Thermal Stability Tests) are described in books of Reilly (1938) and Barnett (1919). These tests are not used in USA

A detailed description of the following stability tests are given by A.J.Clear in PATR **3278** (1965):
75° International Test (p 18),
82.2° C KI Test (p 19)
100° Heat Test (p 19)
90°, 100° and 120° Vacuum Stability Tests (pp 19-22 and Figs 15,16, & 17, pp 46-48)
65.5° Surveillance Test (pp 22 & 23 & Fig 18 on p 49)
80° Surveillance Test (p 23)
120° and 134.5° Heat Tests (pp 23 to 25)
Taliani's Test (pp 25 to 27 & Fig 19 on p 50)
65.5° KI Test (pp 27-29 & Fig 20 on p 51)

89. *Strength of Explosives.* See Power and Strength of Explosives

90. *Surveillance Tests.* See under Stability and Instability and in PATR **3278** (1965), pp 22 & 23

91. *Sympathetic Detonation.* See Detonation by Influence and also under Booster Sensitivity

92. *Taliani Test.* See Vol **1**, p XXIV & XXV and under Stability

93. *Temperature of Detonation or Explosion.* It is the temperature developed on detonation or expln. It must not be confused with Explosion (or Ignition) Temperature, listed here as Item 11

It is listed in Vol **1**, p XXV in which are 8 refs describing the tests

In Vol **4**, under "Detonation (and Explosion) Temperature Developed On" pp D589-L to D601-L) are described the methods of its calculation and several methods of experimental determinations, which include spectrographic method, optical pyrometer and spectra of luminosity obtd by radiation from detonated charges (pp D592-R to D596-R). On pp D596-R to D597-R are listed the following methods: thermocouples, resistance thermometers, pyrometric cones, photography, X-rays, pyrometers, ionization of gas, electron temperature, band spectra, line reversal, spectral distribution and photomultiplier color pyrometer, image converter pyrometer and image opticon spectograph.

On p D597-R of Vol **4**, is briefly described a "Self-Calibrating High-Speed Photographic Pyrometer" devised by J. Tyroler of PicArsn. On pp D597-R & D598-L is abstracted the paper of F.C.Gibson describing the method developed at BurMines. On p D598-L is briefly described the "Spectroscopic Method" developed in Russia by F.A.Baum et al. On pp D598-R & D600-R Ref 46, is described "Color-Optical Method" developed in Russia by I.M.Voskoboinikov & A.Ya.Apin

94. *Transmission of Detonation.* See Ability to Propogate Detonation in Vol **1**, p VII

95. *Trauzl Test of Lead Block Expansion Test.* See Vol **1**, pp XXV & XXVI

96. *Vacuum Stability Tests.* See Vol **1**, p XXIV and in PATR **3278** (1965), pp 19-22 & Fig 16 on p 47

97. *Vapor Pressure.* Its value, usually expressed in mm of mercury column, gives an idea of volatility, and in some cases of stability. Experimental determination is given in Refs listed in Vol **1**, p XXVI

98. *Velocity of Detonation Tests.* See DETONATION VELOCITY TESTS

98a. *Velocity of Detonation-Brisance Relationship.* See Brisance-Detonation Velocity Relationship

99. *Volatility of Explosives.* See Vol **1**, pXXVI

100. *Volume of Gases Evolved on Explosion or Detonation.* See Vol **1**, p XXVI and its six refs

Explosive Properties and Decomposition of Copper Derivatives of Acetylene Hydrocarbons are described by I.M. Dolgopol'skii et al, JAppl Chem (Russian) **19**, 1281-90 (1946) CA **41**, 6721-22 (1947)

Explosive Properties of Initiating Explosives are discussed by A. Suzuki in J IndExplsSoc Japan **14**, 162-63 (1953) & CA **49**, 11381-82 (1955)

Explosive Properties of Mixtures of Organic Substances with Nitric and Perchloric Acids are discussed by L. Andrussow in Chim et Ind (France) **86**, 542-45 (1961) and in Explosivstoffe **1962**, 153

Explosive Properties, including a Blast Test are discussed by H.L. Herman, PATM **1459** (1964) (Conf)

Explosive PTX-1 and PTX-2. See Explosive Compositions PTX-1 and PTX-2

Explosive PVA-4. See Explosive Composition PV-4

Explosive Reactions. Many chemical reactions are explosive and some of them are dangerous.

For example: 1) P.Nawiasky et al reported in C&EN **23**, 1247 (1945) that extremely brisant expl oils were formed when diazotized 4-chloro-o-toluidine was treated with sodium mono- or disulfide

2) H.H. Hodgson, Chem & Ind **1945**, 362 described explosive reactions of diazonium compd with sulfides of sodium. He claimed that he prepd as early as 1918 a brilliant red solid which exploded violently on rubbing with a glass rod, when he treated diaxotized o-nitroaniline with N disulfide. He also listed several later refs in which expl reactions betn diazotized compd and sulfides were reported. Some of these expl products were known in USA as "explosive diazo-sulfides"

3) A.I. Vogel, "Practical Organic Chemistry", Longmans Green, London (1948), 573 stated that reactions betn Na_2S and some diazonium compds might lead to formation of violently explosive diazosulfides and related compd. The following reactions with Na_2S are known to lead to dangerous explns: diazotized derivatives o-nitroaniline, of m-chloroaniline, of 4-chloro-o-toluidine and of 2-naphthylamine

4) R.G.W.Hollingshead & P.E.Garner, Chem & Ind **1953**, 1179-80 drew attention to previous reports on expl nature of the reaction betn certain diazotized substituted benzenes and Na polysulfides and stated that not all the reactions lead to the formation of such compds. For example, when diazotized anthranilic acid was treated with Na polysulfide (as in common use in the dye industry), no formation of expl products was ever observed. H & G warn, however, that in dealing with other diazonium-polysulfide reactions extreme caution must be observed

5) H .Lindlar, Angew Chem **75**, 297-8 (1963), reported an attempt to destroy left over dimethyl sulfate with concd ammonia resulted in an expln, while portionwise addition of dil ammonia was uneventful

6) K.A.Jensen et al, Acta Chem Scand **18** (3), 825 (1964), reported that the reaction of nitrous acid at 0° with methoxythiocarbonylhydrazine ($H_3CO\,C(S)NH\,NH_2$) gave 5-methoxy-1,2,3,4-thiatriazole S-N=N-N=C (OCH_3), which exploded at room temperature. Higher alkyl derivatives were unstable

7) N.Kharasch, JACS **72**, 3322-3 (1950), reported that 2,4-dinitrobenzenesulfenylchloride, $(O_2N)_2C_6H_3SCl$, will expl if prepd at a temp over 90-100°

8) Anon, Chem Eng News **35**, (47), 57 (1957), reported that benzenesulfenyl chloride itself exploded after months of storage in glass at room temp

9) I.B.Douglass & D. A. Koop, JOC **29** (4), 951-2 (1964), reported that CH_3SOCl exploded after months of storage in a sealed ampoule at room temp, probably because of a slow disproportionation to CH_3SO_2Cl and CH_3SCl, the latter being unstable

10) Highly fluorinated compds are extremely dangerous and may react with things which are normally considered "neutral" outside the field of fluorine chemistry. Consider the following examples, presented in chronological order: a) A.G.Sharpe & H.J. Emeléus, JCS **1948**, 2135-8, reported that BrF_3 reacts violently with NH_4F, NH_4Cl, KBr & KI b) A.Kennedy & C.B.Colburn, JACS **81**, 2906-7 (1959) warned of the expl nature of HNF_2, either solid or liq c) R.N.Haszeldine & F. Nyman, JCS **1959**, 1084-90, reported that chlorotrifluoroethene (chilled) ignites in the dark in an oxygen atmosphere when clorine is introduced, but an expln occurs when bromine is added under the same conditions d) C.B.Baddiel & C. F. Cullis, SympCombust, 8th, Pasadena, Calif **1960**, 1089-95 (Publ 1962), reported that ClF_3 ignites methane at ambient temp when the ratio of the two is 2:3, and at even lower ratios as the temp is raised; ignition of propane occurs at temps 25-35° lower than with methane at a corresponding pressure but is less violent e) E.D.Whitney et al, JACS **86** (13), 2583-6 (1964), reported violent reactions with water and explns with common organic solvents when mixed with any of the following: $KClF_4$, $RbClF_4$, $CsClF_4$, $KBrF_6$,

& $CsBrF_6$ f) K.J.Martin, JACS **87** (2), 394-5 (1965), warned of the expl nature of HNF_2 g) K.I. Metz, ChemEngNews **43** (7), 41 (1965), reported that OF_2 will expl when dispersed on 60-80 mesh SiO_2 when confined in a tube less 5mm ID at a temp exceeding 77°K and a pressure of 254mm Hg h) A.G.Streng and F.I. Metz, in separate articles, ChemEngNews **43** (12), 5 (1965), reported that OF_2 and water is highly expl and that OF_2 dispersed on silica is particularly expl when confined in tubing of, or less than, 3mm in diam i) W.D.English & D.R.Spicer, CryogTechnol **1** (6), 260 (1965), reported that liq F_2 on ice at −320° is impact sensitive and will expl The danger here is in allowing water into cryogenic systems containing F_2

11) Another compd shown to be unexpectedly hazardous is dimethyl sulfoxide $(CH_3)_2SO$ or DMSO. The following chronological reports, some "useful", will serve as examples: a) S.A.Heininger & J. Dazzi, ChemEngNews **35** (9), 87 (1957), reported that DMSO decomposes violently when mixed with acyl halides in general, and these in particular: acetyl, benzenesulfonyl, benzoyl, cyanuric, also thionyl halides, phosphoryl chlorides and PCl_3 b) H. Ratz, Ger P1092358 (1960), reported that 1:1 DMSO:$NaClO_4$ or 1:1:1 DMSO:$NaClO_4$:H_2O forms a solid when melted together which is a suitable matrix for Hexogen or NGu/Al c) F.A.French, ChemEng News **44** (15), 48 (1966), reported that the attempt to dissolve NaH in DMSO at 70° resulted in a sudden rise in temp following an expln just as solution was complete d) G.L.Olson, ChemEng News **44** (24), 7 (1966), reported the same as in the preceding item, except that the temp was only 50° and only half as much NaH was involved - in either case the DMSO being in excess over the NaH for the reaction DMSO + NaH = $Na^+DMSO^- + H_2$ e) G.G.Allen et al, ChemInd (London) **40**, 1706-7 (1967), reported that DMSO explodes with acyl chlorides, aliphatic or aromatic, also $SOCl_2$, $POCl_3$ & P_2O_5 f) J.J.M. Rowe et al, JACS **90** (7), 1924 (1968), reported that DMSO may explode when mixed with HIO_4. The only safe procedure is to add small amounts of HIO_4 to DMSO with good stirring g) H.Dehn, Brit P1129777 (1968) and USP3463684 (1969) reported that DMSO can be made to replace some or all of the six waters of crystalization in iron or aluminum nitrates and perchlorates. The resulting material is an expl whose energy release will depend upon the actual number of waters displaced and the salt involved; the addition of AN improves the energy release; also functional as water displacers are methylethylsulfoxide, ethylpropylsulfoxide, and methyldimethylaminomethylsulfoxide

Explosive (or Detonative) Reactions, Theories of. See under Detonation (and Explosion) Theories in Vol 4 of Encycl, pp D601-D629. The different theories are listed on pp D601 and D602

See also the following refs

Refs: 1) H. Muraour, Bull Fr [4] **39**, 1711-16 (1926) & CA **21**, 1185 (1927) (See also CA **20**, 3815) (The theory of the chemical reaction) 2) H.Muraour, Bull [4] **41**, 620-3 (1927) & CA **21**, 2986 (1927) (Reply to criticism of his theory) 3) N.N.Semenoff ZPhysik **48**, 571 (1928) and his book "Chemical Kinetics and Chain Reactions", Clarendon Press, Oxford (1935) 4) B. Lewis, JACS **32**, 3120–27 (1930) (Chem reaction theory of the rate of expln in detonating gas mixtures) 5) H. Muraour, CR **194**, 280 (1932) (Sur une théorie des réactions explosives) 6) H. Muraour, Bull Fr [4] **51**, 1152 (1932) (Sur une théorie des réactions explosives. Cas particulier des explosifs d'amorçage) 7) S. Roginsky, Jour of Physics (Russ) **1**(5), 640 (1932) (Decompn of expls at low temps) 8) H. Muraour, Chim & Ind **29**, 79T to 86T (1933) (Sur une théorie des réactions explosives. À propos d'un récent travail de S. Roginsky) 9) N.N.Semenoff, ActaPhysicochimica (Russia) **1**, 113 & 131 (1934) (Kinematic of exothermic reactions) 10) A.Schmidt, SS **30**, 33-6, 75-9, 101-4, 145-51 (1935) (On the theory of explosive reactions) 11) K.K.Andreev, Bull Fr [5] **2**, 2128-34 (1935) (On the mechanism of explosive reaction. An attempt to apply the theory of chain reactions to certain phenomena observed in expls) 12) K.K.Andreev & Yu.Khariton, TransFaradSoc **31**, No 168 (1935) (On the mechanism of propagation of reactions by chain) 13) C.N.Hurshelwood, ZAngewChem **49**, 370

(1936) (Explosive reactions) 14)D.A.Frank-Kamenetskii, ZhFizKhim **10**, 365-70 (1939); **16**, 357-61 (1942) & **20**, 729-36 (1943) (Mathematical theory of thermal expln, called "stationary theory of thermal expln") 15)K.K.Andreev, KhimReferatZhur **1940**, No 10-11, p 129 & CA **37**, 1604 (1943) (Mechanism of expl reaction) 16)M.Mucahy & A.Yoffe, Australian ChemInst Jour & Proc **11**, 106-20 (1944) (Chain reactions and gaseous explns) 17)Ibid **11**, 134-46 & 166-74 (1944) (Propagation of gaseous explns. A review with 42 refs) 18)Ibid, **12**, 198-214 (1945) (Initiation of expln in solid explns. A review with 37 refs) 19)Ya.B.Zel'dovich, ZhFizKhim **22**, 27-49 (1948) (Theory of flame propagation) 20)J.A.Nicholls et al, "Detonative Combustion" UnivMichigan Research Inst, Final Rept, Project **M898** (1953) 21) M.A.Cook et al, JChemPhys **24**, 191-201 (1956) (Rate of reaction of TNT in detonation by direct pressure measurements) 22)Dunkle's Syllabus (1957-1958) (See Vol **4** of Encycl, p XLIX): p 126 (Reaction front in detonation); 135-42 (Thermal decomposition of solids) 23)M.A.Cook, "The Science of High Explosives", Reinhold NY(1958), pp 123-42 (Reaction rate in detonation); 174-87 (Thermal decomposition of solids); 386-89 (Thermochemistry of detonation and expln) 24)F.A.Baum, K.P.Stanyukovich & B.I.Shekhter "Fizika Vzryva", Moscow (1959), pp 81-108 (Thermochemistry of explosives) 25)K.K.Andreev & A.F.Belyaev, "Teoria Vzryvchatykh Veshchestv", Moscow(1960), p 49-56 (Thermal expln in gases); p 56–61 (Thermal explosion in solids) 26) Encycl of Expls PATR **2700**, Vol 1 (1960), p A501 (Atomic explns, chain reactions in) 27)F.M.Turner, Edit and others "The Condensed Chemical Dictionary", Reinhold, NY (1961), p 246 (Chain reactions) 28)E.Tell, JChemPhys **36**, 901-03 (1962) (On the speed of reaction at high pressure) 29)Encycl of Expls, PATR **2700**, Vol **2** (1962), p C146-L (Chain reactions) 30)Yu.N.Denisov & Ya.K.Troshin, "On the Mechanism of Detonative Combustion", 8th SympCombstn (1962), pp 600-10 31)A.M.Grishin & D.M.Todes, DoklAkad N **151** (2), 366-68 (1963) & CA **59**, 12585 (1963) (Thermal expln with heat transfer by conversion and conduction) 32)P.G.Ashmore & T.A.B.Wesley, "A Test of Thermal Ignition Theory in Autocatalytic Reactions", 10th SympCombstn (1965), pp 217-26 33)Encycl of Expls, PATR **2700**, Vol **3** (1966), pp C430-L to C433-R (Combustion theories as applied to solid propellants) 34)Encycl of Expls PATR **2700**, Vol **4** (1969), p D503 (Reaction front in detonation); pp D504-L to D505-R (Reaction rate in deton); pp D505-R to D506-R (14 refs) (Reaction zone in detonation); pp D619-L to D622-L (Thermal theories and the thermochemistry in detonation, expln, deflagration and decomposition of expls); pp D731-L to D732-L (Expl deflagration or detonative combustion) 35) B.F. Gray, TransFaradaySoc **65**(6), 1603–22 (1969) (Unified theory of explsn, cool flames & two-stage ignitions) & CA **71**, 14732-33 (1969)

Explosive of Rinkenbach & Carroll. See Explosive Composition of Rinkenbach & Carroll

Explosive RIPE. See Explosive Composition RIPE

Explosive Rivets or Other Fastening Devices. (These devices may include *bolts, boars, pins* and *rods*). In the usual process of riveting, a red hot rivet (which is a short cylindrical piece of steel with a head on one end), is set in a hole drilled (or punched) thru plates required to be joined. This is followed by sharp blows on the headless end of the rivet with a hammer (while the rivet is still hot) until the second head is formed. During this operation the opposite end to that being struck is backed-up by a sledge hammer held against it. This method is slow and cannot always be used; for example, when joining plates which can only be reached from one side

The expl rivets, invented about 1940, are great time savers and can be used anywhere

Essentially the expl rivet is an ordinary rivet having a cavity in the end opposite to the rivet head. The cavity is filled with a small quantity of expl of the initiating type. The rivet (or other fastening device like, bolt,

bar, pin, rod, etc) is driven cold into the hole betn two metal pieces to be joined and then the rivet is heated to the temp required to detonate the expl charge. This crimps the headless end and sets the rivet properly

In the original method for initiating the charge, the head of rivet was heated with a device resembling a soldering iron and heat was transmitted thru the metal of the rivet to the opposite end where it detonated the expl charge. This heating device was improved by L.A.Burrows et al (Ref 3) of the DuPont & Co and it could heat the rivet to 100-150°. This method however, had some disadvantages. Much more heat, for example, was required for bringing about ignition in some cases, where a number of rivets were to be heated and where an oxide coating on the rivet head decreased the rate of heat transfer to the metal surface. Another disadvantage came from the fact that with some expl mixts rivets had to be overheated in order to bring about the detonation and this caused corrosion of the rivet. In order to overcome these disadvantages, Burrows et al (Ref 4) proposed a device in which heating was due to induction by high frequency currents, up to 10000 kilocycles. In order to generate such currents, an electron vacuum tube oscillator was used. Other methods for generating high-frequency currents included spark-gap oscillators, various types of electromechanical oscillators or various types alternators

In cases when electrical current is not easily available, as for example, in field work, ignition can be accomplished by fuse, percussion, or by an especially designed fuel torch with narrow flame, which heats only a very small area of the rivet (Ref 6)

The most convenient method of ignition, however, seems to be by means of an electrical conductor introduced into the cavity of rivet (or other connector element, such as bolt, bar, pin rod, etc), and connected to a source of relatively high voltage electricity. The inserted conductor is insulated except at its terminal point. With this conductor sufficiently close to the bottom of the rivet cavity in the vicinity of the expl chge, the circuit may be closed by throwing of a switch. This will cause a spark to jumb across the gap betn the conductor and the metal of the rivet, thereby bringing about the ignition and deton of the expl chge in the rivet. This method is described more fully in Ref 4 and expls used with this method of ignition are listed in Ref 5

A new type of rivet, Fig Ex21, patented in 1951 by Burrows et al (Ref 9) had a cavity extending longitudinally thru a substantial part of the shank and usually entering from the shank end. The diam of the cavity at the shank end was 0.4 to 0.6 of that of the shank diameter, while at the extended portion it is 0.2 to 0.4. The charge of expl (such as a mixt of Hexanitromannite 27, Tetracene 25 & Al powder 48%) was so loaded that the increment in the main cavity was at least beyond the outer surface of the 2nd sheet to be joined. The portion of expl in

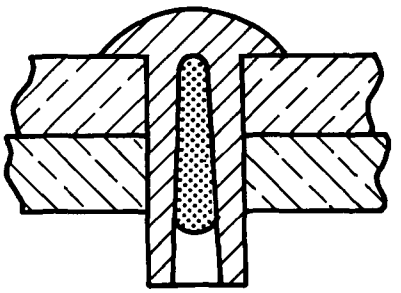


FIG Ex21

reduced diam in the cavity extension served to blast the metal into tight connection with the walls of the hole thru which the rivet was inserted

Numerous compds and compns were proposed for using in expl rivets. One of the earliest compds suggested was LA, but it was found to be unsuitable on account of too high temp of ignition (about 330°). For this reason the Dynamit AG (Ref 1) proposed to lower the flash point by mixing it with one of the following compds: Diazobenzene Nitrate (fl p 90°); Chlorodinitrodiazophenol (fl p 120°); Dibromodiazophenol (fl p 142°); Nitrodiazophenol (fl p 142°); Diazobenzenesulfonic acid (fl p 150°); Silver Acetylide (fl p 150°); Copper Acetylide (fl p 150°) or Guanylnitrosoaminoguanyltetrazene, known as Tetracene (fl p 144°). E.R.von Herz et al (Ref 2) proposed a mixture contg 85-90% of LA with 10-15% of Tetracene. The mixt ignited at 145-150°. Inert substances

could be added to diminish the brisance of the above mixture

Burrows and collaborators of DuPontCo, patented numerous mixtures for use in expl rivets which were suitable for use in expl bolts, bars, pins and rods. It was claimed that expls used in expl rivets, or similar devices, should possess a velocity of detonation above 1000m/sec. To these belong MF, LA, DAzDNPh, Tetryl, PETN, HeN Mannitol, etc. As these expls require fairly high temps for their ignition, easily ignitable expls, such as primary expls: LSt, Cu Acetylide, etc must be incorporated. For example a mixt of LA 70, Tetryl 10 & LSt 20% can be used in expl rivets, fired either by heating method, or by the spark produced by the electrostatic discharge method using a charge preferably below 100000 ergs

In order to determine which expls are capable of ignition by an electrostatic discharge of an energy amounted to 100000 ergs, or less it is necessary to subject each prospective expl to the following "energy requirement test". For this a layer of the expl is spread in thickness of 0.010 inch on a steel plate and is separated by a a short gap from a steel needle. This needle is so arranged that it can be thrown into a closed circuit with a condenser of known capacity, on which a controlled voltage can be imposed. In this way a spark of the desired energy content can be caused to jump the gap from needle to the plate at the locus of the expl layer. Operating accdg to this arrangement, approximate electrostatic energy requirements in ignition of various expls were determined as follows: LSt <500, Cu Acetylide <500, and Pb Nitratohypophosphite <500. Other expls fulfilling the energy requirements are Silver Azide, Basic Lead Picrate and Lead Nitratophosphate. LA required > 200000 energy and although unsuitable by itself could be used in mixts with substances such as LSt

Note: Although most of these expls can be detonated by static charges such as can be accumulated by human bodies, there is no danger of using these expls in metallic rivets. As these rivets are good conductors, no static accumulation is possible in the vicinity of the expl charges

In the last patent before Burrows' death (Ref 9), the following compns for expl rivets are listed:

a) LA 90 & Tetracene 10%

b) HeNMannitol 27, Tetracene 25 & Al powder 48%

With these charges densities of 1.6 to 1.9 g/cc can be satisfactorily used. The amt of expl chge for optimum results will depend on the nature of the expl, the size of the rivet, and the material of which the rivet is constructed. For example, with an Al rivet having a recess of 0.1 inch diameter, a 0.4 grain of LA-LSt would be satisfactory while with larger rivets and the increased size of cavity, as much as 4 grains might be required

For loading a rivet, the expl is introduced into the bottom of the recessed cavity and then the opening is closed. The method preferred by Burrows and collaborators, consists of preparing a jelly by mixing the expl compn with NC gelatinized by a volatile solvent (such as amyl acetate) and then extruding this jelly into place in plastic form in a thin-walled collapsible tube. The extruded mass remains in place where deposited and hardens on subsequent evaporation of solvent. Since hardening or setting requires a certain interval of time, the loading of the rivets should preferably be done prior to their insertion into the objects to be rivetted. In the case of large rivets or fasteners, it is desirable first to charge the expl into a cylindrical metal shell and then to introduce the shell, prior to firing, into the cavity of a rivet. (See also Ref5)

Refs: 1) Dynamit AG BritP 528299 (1941) & CA **35**, 7716(1941) (Detonating charges for expl rivets) 2)E.R.von Herz et al, GerP 702269 (1941) & CA **35**, 8299 (1941) (Detonating charges for expl rivets) 3)L.A.Burrows et al, USP 2295075 (1942) (Portable heating device for firing expl rivets) 4)L.A.Burrows et al, USP 2327763 (1943) (Improved method of firing the expl charge in expl rivets) 5)L.A.Burrows, USP 2387742 (1945)[Several novel types of expl rivets are described. Application of the principle of using expls not only to expl rivets, but to any expl metal connecting element (such as bolts, bars, pins & rods) for fastening one or more articles. Also a list of expl compns used in expl rivets is given] 6)L.A.Burrows

USP 2388901 (1945) (Heating device, resembling torch used by welders, for ignition of expls in rivets) 7)L.A.Burrows & W.E.Lawson, USP 2410047 (1946) (Various expl rivets and other explosively blasting joints) 8)F.F.Huston et al, USP 2412886 and 2412887 (1945) (Application of the principle of expl rivets in the construction of locomotive boilers. It is claimed that completely fluid-tight joints may be produced by expanding the ends of stay bolts thru the use of expl chges) 9)L.A.Burrows & W.E. Lawson, USP 2556465 (1951)(Improvements in explosive rivets and in charges used in them) 10)E.R.von Herz, Explosivstoffe **1954**, 29-38 (Expl rivet called in Ger Sprengniet contained Hexanitromannitol 25, Tetracene 10 & Al powder 65%. Several figs are included in paper) 11)T.Tsukii & S. Kikuchi, Jap P4443 (1954) & CA **49**, 10628 (1955) (Compns used in expl rivets: Diazodinitrophenol 60 and Tetracene 40%; Diazodinitrophenol 60, Tetracene 20, and Hexogen or Nitroguanidine 20%. Oxidizing agents such as $KClO_3$ or KNO_3 may be added) 12)A.H.Holtzman & G.R.Cowan, Ordn **50**,536, March-April 1966 (Explosive rivets were introduced in 1940 for fastening purposes particularly for the aircraft industry. Early expl rivets for aircraft were made of Al; more recently, high temp alloys such as *Inconel X* have been employed)

Note: Inconel X, Trademark of the International Nickel Co. Inc, for an age hardenable wrought alloy conts about 73% Ni, 15% Cr, 0.8% Al, 2.5% Ti and 0.85% columbium

Ref: CondChemDict (1961), 602-R

Explosive "Salex". Accdg to Dr. Herbert Ellern, private communication, Ferguson, Missouri, 30 Dec, 1972 it is a mixture of sulfur and aluminum

Explosive Screw. Buchanan designed & developed a 7/8 inch diam expl screw to meet the following requirements:

a) Must have an external contour in accordance with Drawing PX-8-380

b) Must withstand an acceleration of 5G's

c) Must function satisfactorily at the end of 70 secs exposure to a temp change from ambient to 400°F

d) Must fail at any point on the 21/32 inch diam when assembled in a test fixture

The manufactured screws (600) met the prescribed requirements. The indended application of the Explosive Screw was classified

Ref: E.H.Buchanan, PATR **1769** (1950)(Development of an explosive screw)

Explosive Seam Welding.

A unique, small-scale exp seam welding technique offers improvements over conventional expl welding. Expl welding, accomplished by colliding two metal plates explosively driven together, permits metallurgical bonding that is impossible to achieve by any other joining process, while maintaining material properties. The seam welding technique has successfully joined a variety of aluminum alloys and alloy combinations in thicknesses to 0.125 inch, as well as titanium in thicknesses to 0.056 inch. The explosively welded joints are less than one-half inch in width and apparently have no long-length limitation. The "ribbon explosive" developed in this study contains very small quantities of expl (RDX) encased in a flexible thin lead sheath and initiated with a blasting cap

The evaluation and demonstration of this welding technique was accomplished in three phases: evaluation and optimization of ten major expl welding variables (plate material, plate thickness, explosive quantity: standoff, plate surface, plate deformation, mechanical shock, metal grain orientation, weld length, and expl residual), the development of four weld joints, and an applicational analysis which included photomicrographs, pressure integrity tests, vacuum effects, and fabrication of some potentially useful structures in aluminum and titanium

This joining technique can complement existing fabrication techniques through its simplicity and the ability of producing low-cost joings with strengths up to that of the parent metal. A major disadvantage of this welding technique presently is the reservation in using expls. Other disadvantages are the destructive

mechanical shock produced by the welding operation, and sharp notches at joint interfaces creating stress concentrations. The author is continuing with this work and plans to apply the seam welding technique to a docking system for space stations. He is also interested in hearing of additional applications
Refs: 1)L.J.Bement, "Small-Scale Explosive Seam Welding", Paper presented at the Symposium on Welding, Bonding and Fastening held in Williamsburg, Virginia, May 30-June 1, 1972 2)Gunther Cohn, Edit, Expls & Pyros **5** (7), 1972 (Abstract)

Explosives Sensitivity Data. Card-gap and projectile impact sensitivity data are presented for a wide variety of explosive compositions tested at the Bureau of Mines in more or less standard test geometries. The results of both tests are in good agreement in that they provide the same sensitivity ordering for different subclasses of explosives. Least sensitive were homogeneous liquids that did not exhibit a tendency to undergo low-velocity detonation, Ammonium Nitrate-Fuel Oil, and most cast military explosives. Of intermediate sensitivity were pressed and powdered military explosives, cast Pentolite, permissible and nonpermissible water-based explosives, and one commercial two-component explosive. The most sensitive were permissible and nonpermissible Dynamites and explosives susceptible to low-velocity detonation
Refs: 1) R.W. Watson, "Card-Gap and Projectile Impact Sensitivity Measurements. A Compilation", USBurMines IC **8605**(1973), Order from Publication Distribution Branch, BurMines, 4800 Forbes Ave., Pittsburgh, Pa, 15213. Single copy free 2) G. Cohn, Edit, Expls&Pyrots **7**(1), 1974(Abstract)

Explosive Shattering. See Explosive Disintegration

Explosive Shocks in Air are discussed in the book of G.F.Kinney, "Explosive Shocks in Air", Macmillan, NY (1962). See also definition of Shock in Vol **4** of Encycl, pp D515-R to D517-L and "Detonation, Shock Velocity in Air and Other Substances", pp D529-R to D530-L. Discussion on Shock Wave in Detonation and Explosion is given on ppD530-L to D542-L

Explosive Slurries. Compns consisting of up to 20% water, much AN (dispersed and dissolved), possibly some $NaNO_3$, up to 35% of Al (from shavings down to 30μ in size), a high expl such as TNT, NS, or smokeless powder, perhpas a soluble organic fuel such as sugar, urea glycol, and frequently a natural gum thickener serve to make up expl slurries. An example in Ref 2 is AN 58, Al 32 & water 10%, with a density of 1.7 g/cc and an equivalence in 9 x 40-in size to 55% by weight more of TNT. The more water present the more necessary is a high expl to sensitize the slurry. High deton pressure boosters such as Pentolite or Tetryl are used as detonators
Refs: 1)Cook (1958), 316-21 2)M.A.Cook & H.E.Farnam, Jr, CanP605314 (1960) & CA **55**, 9879 (1961) (Explosive slurries) 3) L.A.Burkardt & W.S.McEwan, USP2992088 (1961) & CA **55**, 26447 (1961) (Explosive Slurry casting) 4)Encycl of Expls, Vol **4**, (1969),p D547-L & R (Detonation in Slurry Explosives)

Explosive Slurry Mixed On-Site in Canal Job. A fluidized explosive, called **Slurrex**, was developed by Hercules Powder Co for widening the Panama Canal's three-mile Gaillard Cut from 300 to 500 feet. The work was supposed to be completed by Dec 1969. Accdg to manufacturer Slurrex enabled blast holes to be filled much faster than by conventional expls. It had also safety advantages, because no expln could take place until all ingredients of Slurrex are blended together and this takes place at blast site just before pumping into the blast holes. Slurrex used for underwater blasting is pumped into polypropylene bags, which resemble 3 to 4 feet sausages. Hercules Co said that precise energy control favors Slurrex,s use in construction, open pit and strip mining

Refs: 1)Hercules Inc, Hercules Tower, Wilmington, Del, 19899 2)G.Cohn, Edit, Expls & Pyrots 1 (8), 1968

Explosive Starter. A device, used to start diesel engines, consists of a ''cartridge-actuated device'' (CAD), inserted in the head of one of the cylinders. On detonation of cartridge a shock (kick)is produced which pushes the piston down and starts the whole engine
Note: CAD's are described in Vol 2 of Encycl, pp C70-R to C72-R
Refs: 1)R.A.Coffman, USP's 2299464, 2299465 & 2299466 (1942) 2)G.Cohn, Edit, Expls & Pyrots 3(1), 1970

Explosive Stimulus Transfer. Under a program to develop a technique to compute probability of detonation transfer between a donor and an acceptor, experiments were performed to measure the effects of seven variables. These included two donor parameters: fragment & slack energy; and five acceptor parameters: confinement, closure thickness, explosive material, explosive particle size and closure material

The donor selected for this study consisted of detonating cord, ferrule & booster. The acceptor expl was confined in either a thin steel cup or a heavy-walled brass bushing. The experimental method consisted primarily of varying a single donor or acceptor parameter, and altering the air gap between the two until an approx 50% fire distance was obtd

For the configurations tested, the following deton transfer principles were indicated:
a) variability in deton transfer is caused by differences in donor output; for a given acceptor design, sensitivity can be treated essentially as constant
b) detonation transfer could not be correlated with the pressure induced in an acceptor expl by donor fragments
c) large variation in extent of acceptor confinement did not appreciably affect deton transfer
d) for a single donor configuration at a fixed air gap, as the acceptor closure thickness was increased, it was necessary to increase acceptor sensitivity in order to maintain the same deton transfer probability
e) tests of two different particle sizes of the same expl showed the coarser material to be more sensitive to shock initiation, and the finer particle size more sensitive to fragment initiation
f) the pressure of any acceptor closure material had an adverse effect on deton transfer

The most significant parameter involved in deton transfer appeared to be the amt of energy induced in the acceptor expl by the donor. Small Scale Gap Test (See Vol 4 of Encycl, p D1085-R) results provided an indication of the amt of energy reqd to initiate an acceptor expl (Ref 1)

The effect on detonation transfer of the parameters shown in Fig Ex22 were reviewed for the donor and acceptor expls RDX, HNS-1 (2,4,6,2′,4′,6′-Hexanitrostilbene of 3 microns particle size) HNS-11 (Same as HNS-1, except of 200-300 microns particle size), DATB (1,3-Diamino-2,4,6-trinitrobenzene), and TATB (1,3,5-Triamino-2,4,6-trinitrobenzene) with a wide cross-section of sensitivities. Eash of these parameters has been individually qualified and explained on both the theoretical level and the impact on practical hardware design. The experimental study is described in Ref 2
Refs: 1) M.L.Schimmel, ''Quantitative Understanding of Explosive Stimulus Transfer (QUEST), McDonnell Douglas Corp Summary Report-Tasks 1 thru 6, **MDC AID21**, St.Louis, Missouri (June 1971) 2)L.J.Bement, ''Quantitative Understanding of Explosive Stimulus Transfer'', Mail Stop 498, NASA Langley Research Center, Hampton, Virginia, 23365 3)G.Cohn, Edit, Expls & Pyrots 5(5), 1972

Explosive Streamers. The design of continuous TNT charges for Mark 29 streamers, carried out at the Explosives Research Laboratory, Bruceton, Pennsylvania, has led to the development of the *Flexed TNT Charges.* The method prepn consists in filling reinforced rubber hose such as of 1 inch diameter

with molten TNT and subjecting the hose, while the TNT is cooling and solidifying, to a mild flexible operation. This produced an easily bending hose contg fine-grained TNT of density 1.4 g/cc which propagated detonation well and was insensitive to rifle bullet impact and to moisture (below 12%)

Six completed Mark 29 Explosive Streamliners, ranging in diameter from 1 to 2½ inches, were assembldd at Yorktown Mine Depot and tested for propagation and damage by countermining. Propagation trials were entirely satisfactory, but countermining trials revealed damage to the outer 2½ inch hose of two streamers

Explosive Streamers are used for mine field clearance and for demolition projects

Ref: J.H.Lum & J.K.Bragg, "The Design of Explosive Streamers" OSRD Rept **1382** (1943), Pertinent to Navy Project NS-125, pp 146 & 378

(Compare with BANGALORE TORPEDOES; Detonating Cables; Demolition Hoses and Demolition Snakes, described in Vol **2** of Encycl, pp B16-R to B18-L

Explosive Strength. See under Explosive Properties

Explosive Substances vs Thermites. Accdg to Schimank et al, *explosive substances* are systems whioh are capable of exothermic chemical change, of a high reaction velocity, and which yield spontaneously gaseous end products wholly or in part

Thermites are systems capable of exothermic chemical reactions, of great (or relatively great) reaction velocity, but not yielding any gaseous end products. Examples of Thermites: Al + Fe_2O_3, Al + $KClO_3$, etc

Refs: 1)H.Schimank, ZElektrochem **24**, 213-14 (1919) 2)W.Bieger & H.Schimank, Ibid, **24**, 354-56 (1919) & CA **14**, 347 (1920) 3) A.Langhans, SS **13**, 310-13 (1918) & CA **14**, 842 (1920)

Explosive Switch. An electric switch operated by an electric squib or primer, such as the MK66 MOD 0. This switch is manufactured by the Atlas Powder Co. The dimensions of the switch housing are ½ x ½ x 2-1/8 inches. It is a single pole quadruple throw switch containing four circuits, two open and two closed. The bridge lead wires extend from one end of the switch a minimum of 4 inches. The bridge wire is nichrome and is approx .001 inch in dia. Its resistance is 1.8 + 0.2 ohms. The ignition spot and base charge consists of LMNR and $KClO_3$ (95-5). The base charge is loaded in two increments, 30 mg at 5000 psi and 25 mg loose

The pressure, exerted against the piston by activation of the squib, overcomes the flange on the piston and drives piston forward which causes the switch slider to move. After traveling a given distance, the slider is locked in place. The locking action takes place when the end contactor in the slider springs into a notch cut into the contact pin holder

Fig Ex 23 is the switch MK66 MOD 0 (Ref 1). There is also given Fig Ex24 of the Switch MK 46 MOD 0 and Fig Ex 25 of its Actuator MK 3 MOD 0

The most recent Army explosive switches consist of the XM60 series (Ref 3). All have six pairs of contacts and measure 3/8 by 3/8 inch. The non-delay switches are 2 inches long while the delay switches are 2½ inches long, the extra length being needed to accomodate the delay column in the squib. The squib charges are similar to those in the MK 66 Mod 0 and the switches look the same except, of course, that there are six contact pairs instead of four. Contacts are double-pole and single-throw with various combinations of normally open and normally closed contacts to permit versatility. The following table lists the available switches while the diagram illustrates a typical contact arrangement

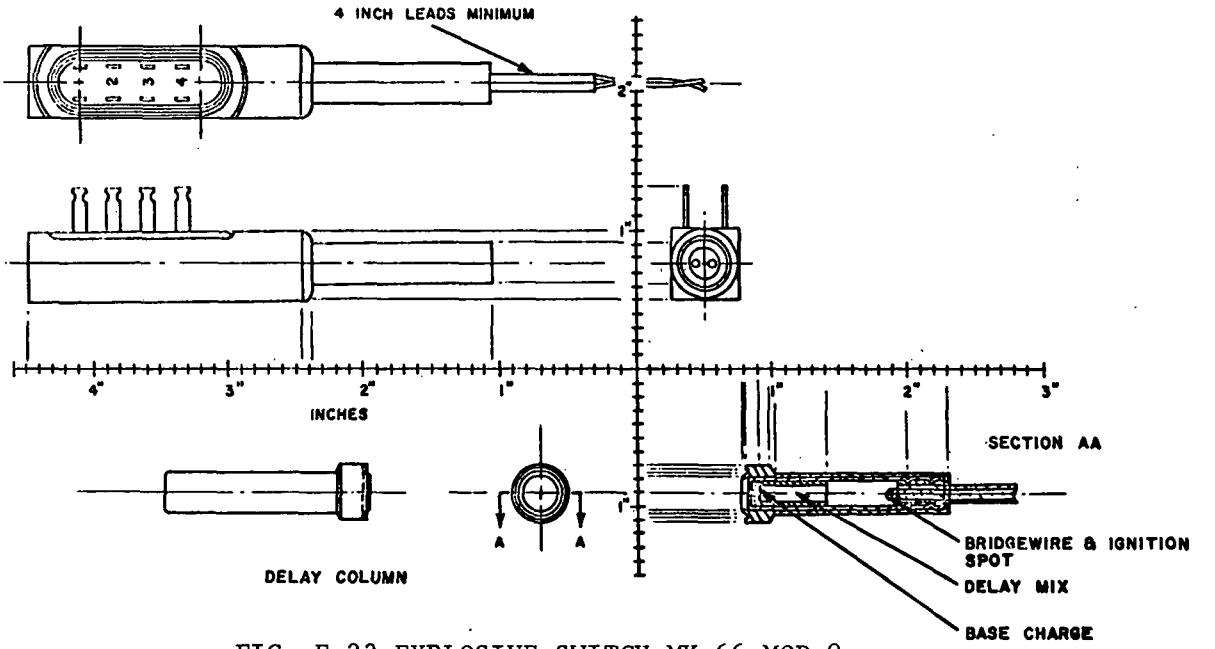
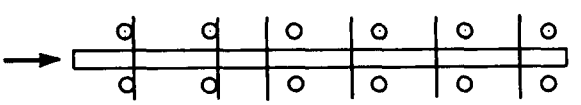


FIG Ex23. EXPLOSIVE SWITCH MK 66 MOD 0

Explosive Switches of XM 60 Series

XM No	Delay	Contacts Closed	Open
60	None	6	0
61	"	2	4
62	"	3	3
63	"	5	1
64	100 Millisec	2	4
65	1 Sec	3	3
66	2 Sec	3	3
67	2 Sec	4	2
68	100 Millisec	4	2

Contact Arrangement of XM 61 Switch



Refs: 1) C.E.Baughn, "Evaluation of Explosive Switches MK66 MOD O and MK 67 MOD O" Nav Ord Rept **6628** (1959), White Oak, Maryland 2)E. Kilmer, "The Actuator, Explosive WOX-23A, An Actuator to Replace Actuator MK 3 MOD O in the Explosive Switch MK 46 MOD O", Nav Ord Rept **6761** (1960), White Oak, Maryland 3) Ellern "Military and Civilian Pyrotechnics", Chem-Publg Co, NY (1968), 168 4)Anon "Fuze Catalog Vol 3 Fuze Explosive Components (U)" **MIL-HDBK-137** (1970)

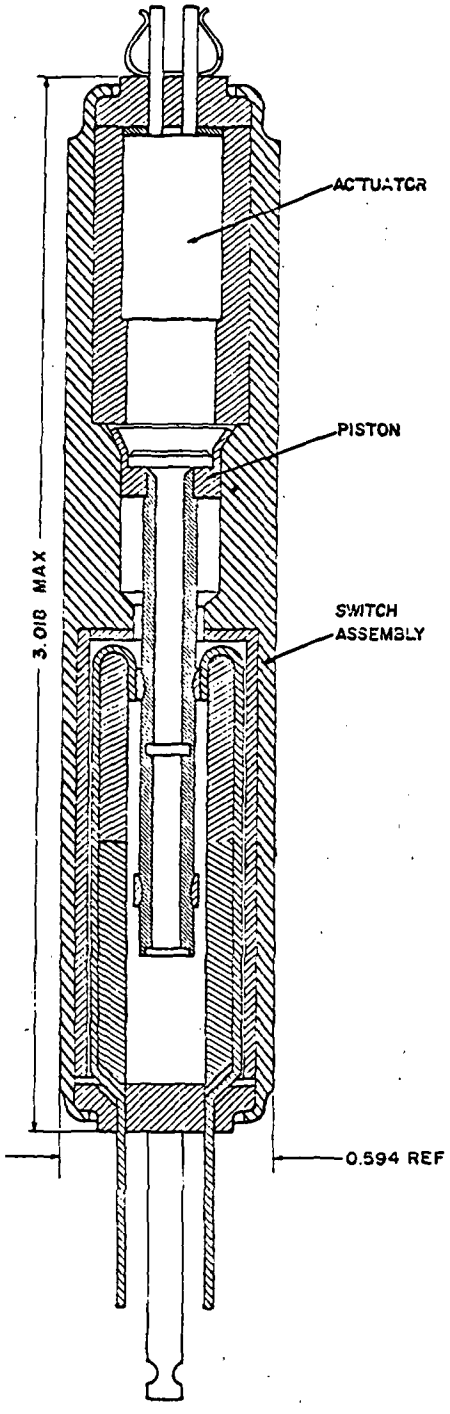


FIG Ex24. EXPLOSIVE SWITCH
MK 46 MOD 0

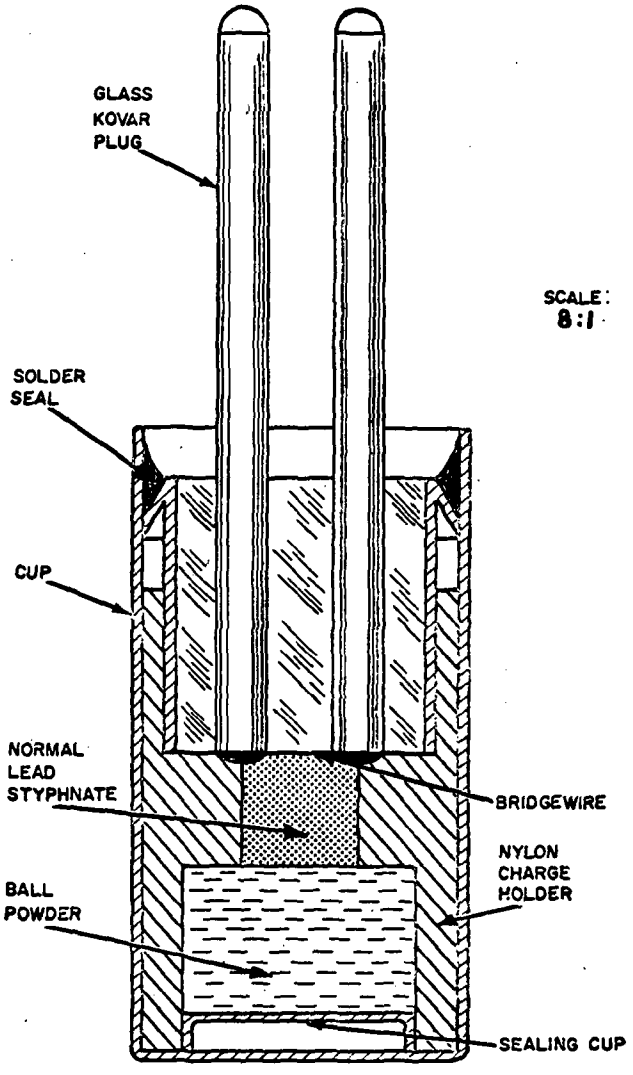


FIG Ex25. EXPLOSIVE ACTUATOR
MK 3 MOD 0

Explosive Systems, Liquid; Sensitivity Characteristics of. The card gap method was used to determine the sensitivity of the following high-energy liq expl systems:
a) Hydrogen Peroxide-Glycerin
b) 50/50 Nitroglycerin-Ethyleneglycol Dinitrate
c) Nitroglycerin-Ethyleneglycol Dinitrate-Triacetin
Further improvements were made to follow continuously the initiation & growth of high velocity detonation, low velocity deton, and low vel reaction. Shock sensitivity scales were established in Al, Plexiglas & polyethylene container. Also several other casting solvents were compared with the above liq expls
Ref: F.C.Gibson et al, "Safety Characteristics of Liquid Explosive Systems" BurMines Progress Rept No 8 (Oct-Dec1963) (Contract IPR19-64-8026-WEPS) and 9, 10 & 11 (1964)

Explosive Train. See Vol 4, pp D836-R to D839-L and Figs 1-21a and 1-21b and the following Refs
Refs: 1) Hayes (1938), 572 2) Ohart (1946) 19-20 3) NOLTR 1111 (1952), 62 4) Anon, "Explosive Trains", AMCP 706-179 (1973)

Explosive Train Test. See Ref 81 in Vol 4, pp D345-L & D345-R. It is an abstract of description given in TM 9-1300-214/TO-11A-1-34 (1967), under the title "Initiating Value Test"

Explosive Test. A normally closed valve opens 3 milliseconds after application of an electrical pulse to a high-temp propellant cartridge. In this one-shot device input from a high pressure line meets a diaphragm, which is too weak to hold off line pressure on its own. Support is provided the opposite face of this diaphragm in the form of a hollow piston, held in place by a shear ring. Upon firing of the propInt, the piston shears the ring and removes the support from the diaphragm. It ruptures allowing the fluid to flow thru the cylinder to a delivery line. The device is capable of operating at fluid pressures of 6500 psig, at temps -65 to 235°F and at altitudes to 200000 feet
Refs: 1) McCormick Selph, PO Box 6, Hollister, Calif, 95023 2)G.Cohn, Editor, Expls & Pyrots 1 (4), 1968 (Abstract)

Explosive Testing in Europe, Standardization of. The 5th Meeting of the European Commission for Standardization Tests of Explosives was held at Dortmund-Derne, Germany on 20–23 October 1971. Forty-two experts from 12 countries attended. Topics discussed included: Lead Block Tests (describing influence of lead quality on test results and manuf of Trauzl Block), Aluminum Block Tests, Detonator Strength Tests, Detonation Through Plastic Disks, Detonation Transfer, and Graded-Strength Blasting Caps for Test Purposes
Refs: 1) Anon, Explosivstoffe 20, pp 81–136 (1972) 2) G. Cohn, Edit, Expls&Pyrots 6(7), 1973

Explosive Wave Shaping by Delayed Detonation was discussed by the late M.M.Sultanoff at the Proceedings of the First Symposium on Detonation Wave Shaping (sponsored by Picatinny Arsenal) at the Jet Propulsion Laboratory, Pasadena, Calif, June 5-7, 1956

Explosive Welding. Accdg to Philipchuk (Ref 1), Davenport & Duvall (Ref 2) lap-type welds of various metals by explosive force have shown experimentally to be entirely feasible. However at the present, it does not appear to be a process which will replace normal welding techniques where a simple weld is adequate and the cost must be kept low. However, it may be used advantageously in the welding of high temperature or refactory metals, metals that are degraded by heat, where large area welds are desirable or where normal welding equipment is difficult to use (See Fig Ex 26)

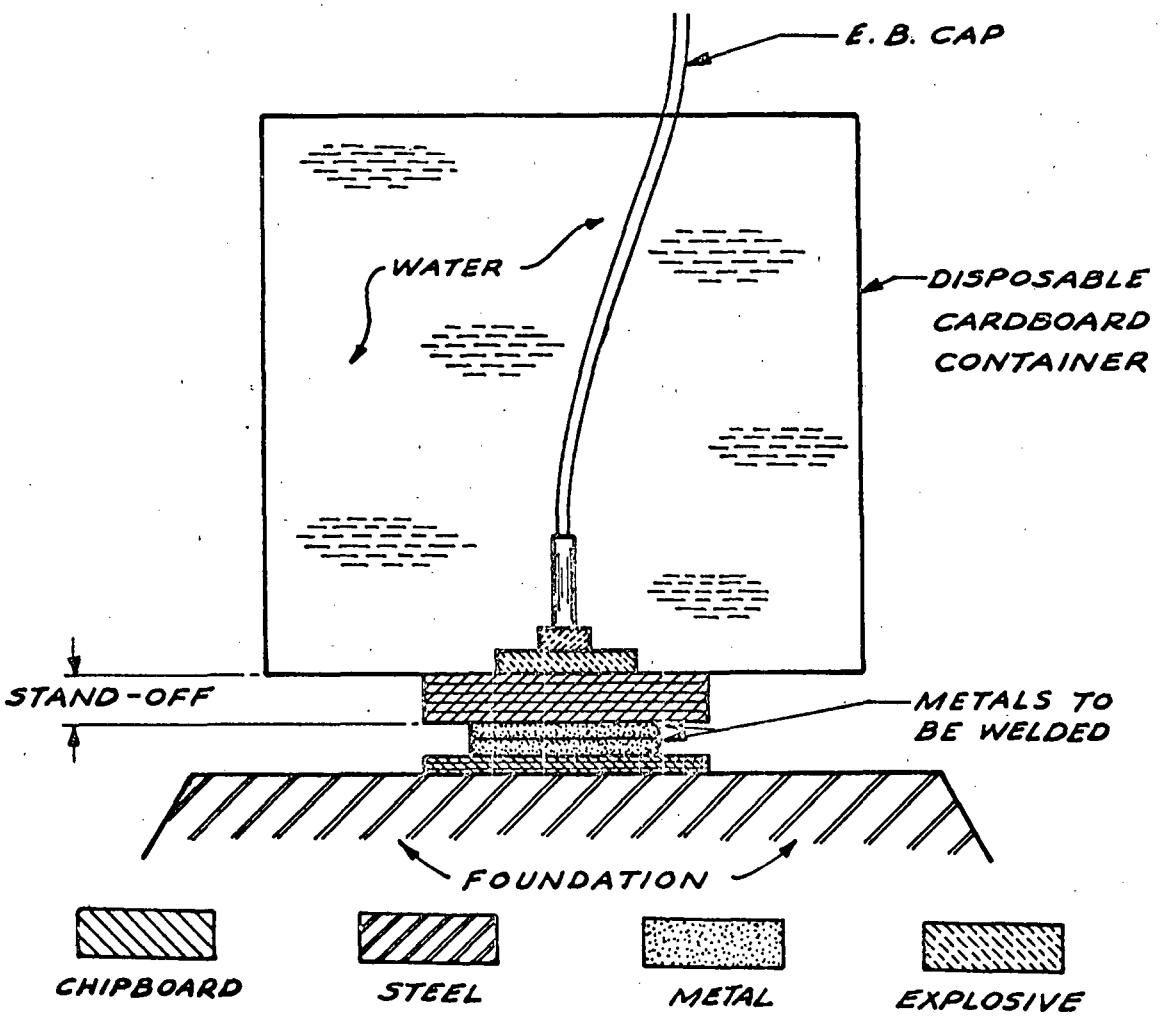


FIG Ex26. TOP INITIATION FOR EXPLOSIVE WELDING

Accdg to Addison et al (Ref 3), expl welding can join many similar and dissimilar material combinations, some of which are often difficult, if not impossible, to weld by the fusion welding processes. With expl process, welding occurs when adjacent surfaces of materials to be joined are properly positioned and thrust together by energy released from an expl source. No external heating is normally introduced in the welding operation, although some heat is generated at the weld interface because of the absorption of energy. Applications of expln welding include: cladding of tubes for reactors; lining of half cylinders for turbine engine applications; cladding of cylinder bores and pistons; welding of pipes and tubing of dissimilar materials for aerospace, cryogenic, and reactor applications; cladding of pressure and mixing vessels for the chemical and petroleum industries, etc. Increasing attention is being given to the welding of tubes to tubeplates for heat exchanger applications

Etzel & Munford (Ref 4) describe an electromechanical device that automatically measures and displays the embedment of each welded joint and locks out the welding machine if the embedment value is not within present upper and lower limits

Lindh & Pershak (Ref 5) describe a semi-empirical method for predicting the effect of weld defects on static and fatigue properties

of weldments and investigated the effects of repair welds on weldments

Pattern (Ref 6) developed a method for producing permanent tube joints that exhibit decreasing leak rate with increasing service temperature

Refs: 1) V.Philipchuk, "Explosive Welding", Aeronautical System Division (ASD) Tech Rept **61-124** (1961), Wright-Patterson Air Force Base, Ohio 2) D.E.Davenport & G.E. Duvall "Explosive Welding", American Society of Tool and Manufacturing Engineers (ASTME) Tech Paper SP **60-161** (1961), Detroit, Michigan 3) H.J. Addison et al, "Explosive Welding of Cylindrical Shapes", Frankford Arsenal, Philadelphia May 1969, 18pp. Order as AD-694359 from National Technical Information Service, P.O.Box 1553, Ravensworth, Virginia 22151 4) J.G.Etzel & J.A. Munford, "Development of a Weld Quality Monitor", NASA Goddard Space Flight Center **N69-33800**, Aug 1969, 11pp 5) D.V. Lindh & G.M. Peshak, "The Influence of Weld Defects and Repair Welds on Performance", Boeing Co, **AD-697 243**, 1969, 66 pp 6) J.W. Patten, "Permanent High Temperature Vacuum Tubing Joints", Boeing Co, **AD-695-493**, 1969, 15 pp 7)Gunther Cohn, Edit., Expls & Pyrots **3** (4), 1970 (Abstract)

"Explosive Working on Metals". Title of the book of J.S. Reinhart & J. Pearson, Pergamon Press, NY (1963)

EXPLOSIVES are solid, liquid and gaseous substances possessing the property, when subjected to either heat, impact, friction or other initial impulse, of undergoing a very rapid exothermic self propagating transformation or decomposition with the formation of more stable materials (usually gases), accompanied by the production of a very loud noise (report) and the development of very great pressures and very high temperatures. This action is called explosion or detonation

The products of expln of compounds containing sufficient oxygen for complete combustion (such as NG) are exclusively gaseous (CO_2 and H_2O); for those containing insufficient oxygen (such as TNT) the gaseous products (CO_2, CO, H_2, CH_4, H_2O etc) may be mixed with some solid particles of carbon (smoke). Still other expls such as Lead Azide, Copper Azide, Silver Fulminate etc, produce some metallic particles in addition to gases. In a few cases, such as with Copper Acetylide, no gases are developed whatever, only solid particles of C and Cu; it seems that any expl value of such materials depends on the heating effect on adjacent gases. The gaseous products of expln generally have a volume much greater than that of the expl itself. The work done by an expl is determined primarily by the amount of heat given off during the expln. An important characteristic of nearly all expls (except Initiating Compounds) is their oxygen balance (See under Available Oxygen in Vol 1, p 515)

Solid and liquid expls can be divided into two classes:

A) Low Explosives (L E) or Propellants and

B) High Explosives (H E)

A) Low Explosives, such as Black Powder or Smokeless Propellant when used as propulsive charges, undergo deflagration (rapid burning) at rates ranging from centimeters to meters per second. This is accompanied by the development of large volumes of gases, high temperatures and pressures, but no real expln takes place- the gases simply push the projectile out of the weapon (propulsive action). They also can be used as commercial Blasting Explosives

B) High Explosives are those which undergo detonation, or explosion in a small fraction of a second

High Explosives (HE) can be divided into HE proper and Initiating Explosives. In our opinion, this classification is inadequate because it overlooks a class of expls (such as commercial Blasting Powders) which cannot strictly be called HE because they are of comparatively low brisance and velocity of detonation. They possess "heaving action" and are not suitable as bursting charges in projectiles or for blasting operations in hard rock. For this reason we would like to propose the following subdivision of HE's:

1) Priming (or Primary) Explosive Compounds or Mixtures (such as Lead Dinitroresorcinate and Lead Styphnate) are sufficiently sensitive to impact or friction to explode (or deflagrate), developing a high temperature and producing a flame, but no real detonation. They are used as *priming (top)* charges in compound detonators

2) *Initiating (or Detonating) Explosives* are sufficiently sensitive to heat, impact or friction to undergo detonation and to transmit the detonation wave to less sensitive expls as described below. As a rule these expls are less brisant and less powerful than HE, but there are some exceptions, notably Cyanuric Triazide. Mercuric Fulminate (MF) had been used until recently in single component detonators but now it is nearly completely replaced by Lead Azide (LA) which is used as *base (detonating) charge* in compound detonators. As their top charge Lead Styphnate (LSt) is used

(See DETONATORS, IGNITERS AND PRIMERS in Vol 4, pp D733-L to D743-R)

3) **High Explosives** differ from primary and initiating expls in not being exploded as easily by impact, friction or heat. They may be divided in the following subgroups:

a) *Highly Brisant and Sensitive HE's* such as Blasting Gelatin and 60% Gelatin Dynamite, possess high brisance and power, but are too sensitive to be used for military purposes as boosters or bursting charges in shells. They can, however, be used as demolition charges or for blasting hard rocks. Their detonation velocities can be approx 7500 to 8500 m/sec

b) Booster Type HE's, such as straight Tetryl, PETN or RDX are very brisant and powerful expls, less sensitive to mechanical shock than a, but too sensistive to be used as bursting charges in shells but suitable as booster charges. Their detonation velocities can be approx 7000 to 8000 m/sec

c) Bursting Type HE's such as TNT, Pentolite, Composition B or Tetrytol are brisant and powerful, and are sufficiently insensitive to shock to be used as bursting charges in shells. They are very difficult to detonate directly by detonators or blasting caps (unless they are very large ones) and for this reason boosters are used as intermediate charges in order to insure complete detonation. The detonation velocity of bursting type HE's are approx 7000 m/sec. Cannot be used for blasting soft coal or ground but can be used for blasting hard rock

d) Commercial Blasting Explosives, such as Low and Dynamites (not higher than 40%), Permissible Explosives and Black Powder (when used as an expl and not as a propellant), are fairly powerful but of very low brisance and velocity of detonation (such as 300 to 3000 m/sec). Their action is *propulsive (heaving),* caused by hot gases which are evolved on expln. They are used for blasting soft rock, the ground, or coal, but cannot be used successfully in modern shells because most of their energy would be dissipated in rupturing the walls of the shells and their fragments would be too big and too few. Blasting expls are usually less expensive than military expls and this is the important reason for their commercial use

Refs: 1) Hayes (1938), 1-9 2) Davis (1943), 1-6 3) Ohart (1946), 16-19 4) Kirk & Othmer, Vol 6, 1st edition (1951), pp 1-91, article by W.H. Rinkenbach et al, Vol 8, 2nd edit (1965), pp 581-658

EXPLOSIVES (LISTED IN PREVIOUS VOLUMES OF ENCYCLOPEDIA).

As a rule explosives were listed and described under their parent compounds arranged in alphabetical order, as for example 2, 4, 6-Trinitroaniline or Picramide is described under ANILINE in Vol 1 of Encycl. Most of the metallic salts are described under the names of their anions as, for example, Lead Azide (LA) is described under AZIDES, Mercuric Fulminate (MF), under FULMINATES, whereas some salts are described under the names of their cations, as for example Ammonium Nitrate (AN) is described under AMMONIUM in Vol 1

As an exception there are listed in several volumes, explosives, not completely des-

cribed but with listed important explosive properties, such as shown in the following examples;
1) Vol 2, p B29 - Belgian Industrial Explosives
2) Vol 2, pp B202-R to B203-R - Blasting Explosives
3) Vol 2, p B211-R - Blasting Gelatin
4) Vol 2, pp B266 to B297 in Table I, under BRISANCE are listed about 340 explosives with their Brisance and Power, whereas in Table II on p B298 are listed Detonation Velocities of 21 explosives
5) Vol 2, Tables 1 to 6, pp C156 to C159 are listed CHEDDITES
6) Vol 3, under COMMERCIAL EXPLOSIVES are listed on pp C437-L to C444-R "Coal Mining Explosives, Nonpermissible", while on pp C444-R to C456-R are "Coal Mining Explosives Permissible"
7) Vol 5, pp D1584-L to D1742-R are described DYNAMITES

EXPLOSIVES (BOOKS AND PAMPHLETS Listed in Chronological order)

1) J.P.Cundill, "A Dictionary of Explosives" London, 1889, The French translation was published in MP 5, 235-354 (1892) & 6, 7-132 (1893)
2) O.Guttman, "The Manufacture of Explosives", Whittaker, London (1895) 2a)S.G. Romocki, "Geschichte der Explosivstoffe", Berlin & Hannover, Vol 1 (1895) & Vol 2 (1896)
3) J.Daniel, "Dictionnaire des Matières Explosives", Dunod, Paris (1902)
4) A. Noble, "Artillery and Explosives", Murray, London (1906)
5) L. Gody, "Traité Théorique et Pratiques des Matières Explosives", Wesmaël-Charlier, Namur (1907) 5a) R.Escales, "Die Explosivstoffe", Veit & Co, Leipzig (1908 - 1926), which includes: I.Schwarzpulver und Sprengsalpeter; II. Die Schiessbaumwolle; III. Nitroglyzerin und Dynamit; IV.Ammonsalpetersprengstoffe; V. Chloratsprengstoffe; VI. Nitrosprengstoffe; VII. Initialexplostoffe; VIII. Das rauchlose Pulver
6)P. F.Chalon, Les Explosifs Modernes, "Ch. Béranger, Paris (1911) 6a) G.W.MacDonald, "Historical Papers on Modern Explosives", Whittaker, London & NY (1912)
7) W.R.Quinan, "High Explosives", Crichley-Parker, London (1912)
8) H. Brunswig, "Explosives", Wiley, NY (1912) 8a) L. Vennin et G.Chesneau, "Les Poudres et Explosifs", Ch. Béranger, Paris (1914)
9) E.M.Weaver, "Notes on Military Explosives", Wiley, NY (1917) 9a) A.F.Ramsey & H.C.Weston, "Manual on Explosives", London (1917)
10) A. Marshall, "Explosives", Churchill, London, Vol 1 (1917) & Vol 2 (1917)
11) E.de W.S.Colver, "High Explosives", Van Nostrand, NY (1918)
12) E. de B.Barnett, "Explosives", Van Nostrand NY (1919)
13) P. Verola, "Chimie et Fabrication des Explosifs", A. Colin, Paris (1920)
14) A. Marshall, Dictionary of Explosives, Churchill, London (1920)
15) H.Kast, "Spreng-und Zündstoffe", Vieweg, Braunschweig (1921)
16) E.A. Evieux, "Artifices, Poudres et Explosifs", Baillière, Paris (1923)
17) Ph. Naoúm, "Schiess-und Sprengstoffe", Steinkopf, Dresden (1927) 17a) A.P. VanGelder & H. Schlatter "History of Explosives Industry in America, Columbia UnivPress, NY (1927)
17b) Ph. Naoúm, "Nitroglycerine and Nitroglycerine Explosives", translated from Ger by E.M. Symmes, Williams & Wilkins, Baltimore (1928)
18) R. Molina, "Esplodenti e Modo de Fabricarli", Hoepli, Milano (1930)
19) P. Pascal, "Esplosifs, Poudres, Gaz de Combat, Hermann, Paris (1930) 19a) R. Assheton, "History of Explosives on Which the American Table of Distances was Based", C.L. Story Co, Delaware Press, Wilmington, Del (1930)
20) L.Vennin, E. Burlot et H. Lécorché, "Les Poudres et Explosifs", Ch. Béranger, Paris (1932)
21) A. Stettbacher, "Die Schiess- und Sprengstoffe", Barth, Leipzig (1933)

22) J. Pepin Lehalleur, "Traité des Poudres, Explosifs et Artifices", Baillière, Paris (1935)
23) C. Beyling & K. Drekopf, "Sprengstoffe und Zündmittel", Springer, Berlin (1936)
24) J. Reilly, "Explosives, Matches and Fireworks", Van Nostrand, NY (1938)
25) K.K. Snitko & M.A. Budnikov, "Vzryvchatyiye Veshchestva", Artilleriiskaya Akademia, Moscow (1939) 25a) E. Piantanida, "Chimica degli Esplosivi e dei Gas di Guerra", Tipo-Litografia della Accademia Navale, Livorno (1940)
26) A.G.Gorst, "Khimia i Teknologia Nitrosoyiedinenii" (Chemistry and Technology of Nitrocompounds), Oborongiz, Moscow (1940)
26a) "Thorpe's Dictionary of Applied Chemistry", Vol 4 (1940)
27) E.E.Sancho, "Química de los Explosivos", Aguado, Madrid (1941) 27a) V. Mathesius, "Zur Geschichte der Sprengstoffe", Hoppenstedt, Berlin (1941) [Reviewed in SS 37, 111-12 (1942)]
28) J. Bebie, "Manual of Explosives, Pyrotechnics and Chemical Warfare Agents", Wiley, NY (1943)
29) T.L.Davis, "The Chemistry of Powder and Explosives", Wiley, NY (1943)
30) M. Meyer, "Explosives", Crowell, NY (1943)
31) G.D.Clift & B.T. Fedoroff, "A Manual of Explosives Laboratories", Lefax, Philadelphia, Vols 1-4 (1942-1944)
32) A.L. Olsen & J.W.Greene, "Laboratory Manual of Explosive Chemistry", Wiley, NY (1943)
33) H. Kast & L. Metz, "Chemische Untersuchung der Spreng- und Zündstoffe", Vieweg, Braunschweig (1944)
34) H. Blatt et al, "Compilation of Data on Organic Explosives", OSRD Rept 2014 (1944)
35) M. Vivas, R. Feigenspan & F. Ladreda, "Pólvoras y Explosivos Modernos", J. Morata, Madrid, Vols 1-5 (1944-1948)
36) A. Pérez Ara, "Tratado de Explosivos", Editorial Cultural, La Habana (Cuba)(1945)
36a) "The Preparation and Testing of Explosives", Summary Tech Rept of Div 8, NDRC, Vol 1 (1946)
37) N.A.Shilling, "Vzryvchatyiye Veshchestva i Snariazheniye Boyerpripassov" (Explosives and Ammunition Loading), Obononghiz (Moscow) (1946)
38) H.Muraour, "Poudres et Explosifs", Presses Universitaires, Paris (1947)
39) A. Mangoni, "Quaderni di Chimica Industriale No 14, Esplosivi", Pàtron, Bologna (1947)
40) A. Stettbacher, "Spreng- und Schiesstoffe", Rascher, Zürich (1948)
41) Col Camillo Caprio, "Corso di Esplosivi", Scuola Salesiana del Libro, Roma, Vol 1 (1948); Vol 2 (1949)
42) M.Giua & C. Giua - Lollini, "Dizionario Li Chimica Generale e Industriale", UTET, Torino, Vols 1-3 (1948-1950)
43) T.Tharaldsen, "Eksplosivstoffer", Dreyers Verlag, Oslo (1950)
44) J.M.Jiménez, "Explosivos", Ediciones Ejército, Madrid (1951)
45) C. Belgrano, "Gli Esplosivi", Hoepli, Milano (1952)
46) A. Stettbacher, "Pólvoras y Explosivos", G. Gili, Buenos Aires (Argentina) (1952)
47) J. Taylor & E.Whitworth, "Explosives", pp 524-538 with 207 refs on the book "Reports on the Progress of Applied Chemistry", Vol 37, published in 1952 by the Society of Chemical Industry in London
48) B.Wiesner, "Der Verkehr mit Sprengstoffen" (Intercourse with Explosives), E.Barth, Manheim (1952)
49) F.Weichelt, "Hanbbuch der gewerblichen Sprengtechnik", C. Marhold, Halle (1953)
50) H. Gilman, Editor, "Organic Chemistry", Vol 4 (1953), Chapter on Chemistry of Explosives, pp 952-1000 by G.F.Wright
51) L.V.Dubnov, "Predokhranitel'nyiye Vzryvchatyiye Veshchestva" (Permissible Explosive Substances), Obronghiz, Moscow (1953)
51a) B.Rowland & W.Boyd, "US Navy Bureau of Ordnance in WWII", US GovtPrinting Off, Washington, DC (1953), 539 pp
52) D.Smolenski, "Teoria Materialów Wybuchowych" (Theory of Explosive Substances), Wyd MinistOborony Narodowej, Warsaw (1954)
53) G.Castellfranchi & P. Malatesta, "Lezioni di Chimica di Guerra", Ed Studium, Roma

(1954), Part 1 "Esplosivi", Part 2 "Aggressivi Chimica (1954)
54) H.E.Watts, "The Law Relating to Explosives", Griffin, London (1954)
55) T.Urbański, "Chemia i Technologia Materialów Wybuchowych", Wydawnictwo MON (1954)
56) Ph. Naoúm & A. Berthmann, "Explosivstoffe, Sprengmittel, Treibmittel oder Schiessmittel und Zündmittel", Manser, München (1954)
57) E.M. González, "Explosivos", Ediciones Ejército, Madrid (1955)
57a) B.T.Fedoroff et al, "Dictionary of Russian Ammunition and Weapons", PATR 2145 (1955)
58) Anon, Military Explosives; TM9-1919 (1955)(See its Revision Ref 89)
59) M.A.Budnikov, A.A.Levkovich & I.V.Bystrov, "Vzryvchatyiye Veshchestva y Porokha" (Explosive Substances and Propellants), Oboronghiz, Moscow (1955)
60) A.G.Gorst, "Porokha y Vzryvchatyiye Veshchestva" (Propellants and Explosives), Oboronghiz, Moscow (1955)
61) N.E. Yaremenko & B.Ya.Svetlov, "Teoriya i Teknologhiya Promyshlennykh VV" (Theory and Technology of Industrial Explosives), Promstroyizat, Moscow (1957)
62) K.K. Andrejew (Andreev), "Sprengstoffe", Verlag des Ministeriums für Nazionale Verteidigung, Berlin (1957)
63) J. Taylor & P.F.Gray, "British Coal Mining Explosives", Newness, London (1958)
63a) R.McAdam & R.Westwater, "Mining Explosives", Oliver & Boyd, Edinburgh (1958)
64) B.T.Fedoroff et al, "Dictionary of Explosives, Ammunition and Weapons"(German Section), PATR 2510 (1958)
65) W.R.Tomlinson, Jr & O.E.Sheffield, "Properties of Explosives of Military Interest PATR 1740, Revision 1 (1958)(See also Ref 88)
66) M.A.Cook, "The Science of High Explosives", Reinhold, NY (1958)
67) T.Urbański, "Chemie a Technologie Výbusin" (Chemistry and Technology of Explosives), StátniNakladelstvíTechnické Literatury, Praha, Vol 1 (1958)
68) M. Giua & M.L.Marchino, "Esplosivi", pp 1 to 497 in M. Giua, "Trattato di Chimica Industriale", UTET, Torino VI (1)(1959)
69) W.Taylor, "Modern Explosives", Royal Institute of Chemistry, Monograph No 5, London WCI (1959)
70) Ye.Yu.Orlova, "Khimiya i Tekhnologhiya Brizantnykh VV" (Chemistry & Technology of High Explosives) GosNauchTekhnIzdat, 2 parts (1960)(Engl transln in Technical Documentation Liaison Office, Wright, Patterson Air Force Base, Ohio, MCL-844/M2,
70a) R.P.Antonelli, "Encyclopedia of Explosives", OTIA, Ordnance Liaison Group-Durham North Carolina (1960)
71) Anon, "Analytical Methods for Powders and Explosives", AB Bofors Nobelkrut, Sweden (1960)
71a) C.E.Gregory, "Explosives for Engineers" A Primer for Australian Industrial Practice, Univ of Queensland Press, Brisbane (1960)
72) T. Urbański, "Chemie und Technologie der Explosivstoffe", VEB-Deutscher Verlag für Grundstoffindustrie, Leipzig, Vol 1 (1961), Vol 2 (1963) & Vol 3 (1964)(Original is in Polish)
73) F. Weichelt, "Handbook der Sprengtechnik, ", Dentscher Verlag, Grundstoffind, Leipzig (1961)
75) H.Koenen & H. Freytag, "Definitions for Explosive and Similar Materials", TIL/T. 5193 (1962)
76) J. Berger & J. Viard, "Physique des Explosifs Solides", Dunod, Paris (1962)
77) B.I. Belkovski & G.T.Dyakov, "Vzrivni Veshchestva Teoriya, Khimiya, Tekhnologiya i Upotreba" (Explosive Substances: Theory, Chemistry, Technology and Use), Tekhnika, Sofia (Bulgaria)(1962)
77a) K.K.Andreev et al, "Teoriya VV" (Theory of Explosives), Oboronghiz, Moscow (1963)
78) V.A. Assonov, "Vzryvchatyiye Veshchestva in Ikh Primeneniye v Gornoy Promyshlennosti" (Explosives and Their Use in Mining Industry), GosNauchinTekhn Izd LitGornDelu, Moscow (1963)
79) R.H. Pierson, "Explosives and Propellants", Analytical Procedures, pp 1283 to 1410 in "Standard Methods of Chemical Analysis", Vol 2 Part B (1963), E.J. Welcher, Edit, Van Nostrand, Princeton, New Jersey

80) E.L. Gómez, "Estudio de los Explosivos Industriales", Madrid, reviewed in Explosivstoffe **1964**, 126
81) M. Novotný & V. Pesata, "Teorie Prumyslových Traskavy" (Theory of Industrial Explosives), StatniNaklTechLit, Praha (1963)
82) T.Urbański, "Chemistry and Technology of Explosives", Pergamon Press, NY, Vols **1-3** (1964,1965,1967) (Translated from Polish)
82a) A.A. Shidlovskii, "Osnovy Pirotekhniki" (Fundamentals of Pyrotechnics), Oboronghiz, Moscow (1964) [Engl translation by US Joint Publications Research Service, PATM **1965** (1965]
83) Wm.H. Rinkenbach, "Explosives", pp 581 to 658 in Kirk-Othmer's, "Enclopedia of Chemical Technology", Vol **8**(1965), Interscience Publishers, NY, 28 refs
84) R.R. Lenz, "Explosives and Bomb Disposal Guide", Thomas, Springfield (1965)
84a) A.P. Cartright, "The Dynamite Company The Story of African Explosives and Chemical Industries Limited", Macdonald, London (1965)
85) B.Ya. Svetlov & N.E. Yaremenko, "Teoriya i Svoystvo Promyshlennykh VV" (Theory and Properties of Industrial Explosives", Nedra, Moscow (1966)
86) S. Fordham, "High Explosives and Propellants", Pergamon Press, NY (1966)
87) E.I. duPont de Nemours, "Blasters' Handbook", Wilmington, Delaware (1966), pp 25–8 (Black Powder); 29–46 (Dynamites); 47–68 (Blasting Agents); 68–80 (Water Gel Explosives); 81–86 (Jet Tappers); 87–110 (Initiating Devices)
88) W.R. Tomlinson & O.E. Sheffield, "Properties of Explosives of Military Interest", Army Materiel Command Pamphlet, AMCP **706–177**, March 1967 (Revision of PATR **1740**, listed as Ref 65)
89) Anon, "Military Explosives", **TM 9–1300–214** & **TO 11A–1–34**, Nov 1967 [Revision of **TM 9–1910** (1955), listed as Ref 58]
90) P. Tavernier, "Poudres et Explosifs" (Propellants and Explosives), PressesUniversitaires, Paris (1969)
91) F.A. Baum et al, "Termostoykiye VV i Ikh Deystviye v Glubokikh Skvazhinakh" (Thermally Stable Explosives and Their Action in Deep Boreholes", Nedra, Moscow (1969)
92) C.H. Johansson & P.A. Perrson, "Detonics of High Explosives", Academic, London (1970), Reviewed in Expls&Pyrots **5**(3), March 1972, The Franklin Institute
93) F. Pristera (Picatinny Arsenal), "Explosives", pp 405–71 with 67 refs in "Encyclopedia of Industrial Chemical Analysis", Vol **12**, Wiley, NY (1971) (Analyses and properties of common Primary and High Explosives)
94) B.D. Rossi & Z.G. Pozdnyakov, "Promyshlennyie Vzryvchatyiye Veshchestva i Sredstva Vzryvaniya Spravochnik" (Industrial Explosives and Initiating Agents Handbook), Nedra, Moscow (1971)
95) A.G. Gorst, "Porokha i Vzryvchatyiye Veshchestva" (Propellants and Explosive Substances), Izdat "Mashinostroyeniye", Moscow (1972)
96) N.J. Blay & I Dunstan, "Compatibility and Stability Testing of Explosives and Solid Propellants", US NTIS, AD Rep **1970**, No 744871, 28pp

The following complimentary copies were received from Dr O. Vettori of Aulla (Massa C), Italy:

A) Pamphlet: "Sorlini Esplosivi SPA", Brescia, N. Italy
B) Pamphlet: "Esplosivi Mangiarotti", Codroipo (Udine) (1959)
C) Book: C. Giorgio, "Tecnica degli Esplosivi", DelBianco-Udine (1964)
D) Book: E. Brandimarte (Accademia Navale), "Le Cariche di Scoppio" (Bursting Charges), Poligrafico dell' Accademia Navale, Livorno, Italy (1966)
E) M. Busco, "Ottica Geometrica degli Esplosivi", Vol 1 (Geometric Optics of Explosives), Published by Author, Vile Quatro Venti, 247-Roma (1973)
F) M. Busco, "Ottica Fisica degli Esplosivi" (Physical Optics of Explosives), Roma (1973)

EXPLOSIVES OF MILITARY INTEREST
(Papers and Reports Listed in Chronological Order)

In addition to references listed under individual explosives in Vols **1**, **2**, **3**, **4** and **5**, the following may be included:

1) J.D. Hopper, "Study of Fundamental Properties of High Explosives", PATR **838**(1937)

2) E. von Herz & Ph. Naoúm, GerP 659721(1938) (A moldable methyloxamide and nitrocompds used for making charges such as with TNT, TNNaphthalene, etc)

3) G.C. Hale, USP 2202851(1940) & CA **34**, 6815(1940) (Explosive suitable for bombs and shells, such as consisting of PA 85–90 & MNNaphthalene 15–10%)

4) J.D. Hopper, PATR **1041**(1940); **1063**(1940) and **1133**(1941) (Explosives of High Brisance)

5) D.D. Sager, PATR **1155**(1942) (Explosives of High Brisance)

6) W.R. Tomlinson, Jr, PATR **1170**(1942) (Explosives of High Brisance)

7) R.H. Kent, "Explosives and Their Military Application", JApplPhys **13**, 348–54(1942) [Definition is given of terms: Sensitiveness to Impact & Friction (p 348), Force (p 349), Rate of Detonation (p 350), Density (p 351), Pressure (p 352), Energy (p 352), Blasting Power (p 352), Brisance or Shattering Power (p 352), and Phenomena Attending an Explosive (pp 353–54)]

8) A. Stettbacher, Protar **8**, 81–92(1942) & CA **37**, 1603(1943) [Theoretical discussion based upon the following values: density (d), gas vol per kg of expl (V_o), heat of explosion (Q), temp of explosion (T) and the sensitiveness to impact (S). The opinion is expressed that explosive technique cannot find anything new because all possibilities have been explored, and further advance must consist in finding new methods of use]

9) Schweiz Sprengstoff-Fabrik AG, SwissP 226852(1943) & CA **43**, 2438(1949) (Highly brisant expl of low sensitivity to shock consists of PETN desensitized by a mixture of paraffin and nitropentaerythritol in which at least 3 OH groups are esterified with stearic acid)

9a) A.H. Blatt, "Compilation of Data on Organic Explosives", OSRD Rept **2014** (1944), Typewritten book published by Ordnance Dept, Washington, DC

10) W.R. Tomlinson Jr, PATR **1360**(1943); **1379**(1944); **1402**(1944); **1414**(1944); **1429** (1944); **1437**(1944) and **1452**(1944) (Explosives, Ternary Mixtures)

11) A.J. Phillips, PATR **1445**(1944) (Properties of Ternary Mixture of Explosives. The Manufacture of PTX-2 by the Slurry Precipitation Process)

12) H.A. Aaronson, PATR **1469**(1944) (Properties of Ternary Mixtures of High Explosives)

13) Wm. H. Bagley Jr, USP 2369517(1945) & CA **39**, 3162(1945) [Explosive suitable for small-caliber projectile comprises a highly electropositive material such as $Pb_3(OH)_4(ClO_4)_2$ 84% and a highly electronegative material such as powdered Al 16%, both of 200–400 mesh granulation. The mixt is compressed to about 1/3 its vol. An alloy of Al and Ca, or straight Mg may be substituted for the Al, and $Cu_3(OH)_4(ClO_4)_2$ for the basic Pb salt]

14) H.L. Porter, CIOS **33–27**, pp 6–46(1945–1946) (Explosives, Hollow Charge and Shock Waves) [Description of work at the Physics and Ballistics Institute, Air Ministry, Berlin, under Director Dr H. Schardin is given. It was claimed that Dr Max von Förster had shown in 1883 (earlier than Ch.E. Munroe) that bare hollow charges gave an enhanced effect along the axis of the charge

The following persons, besides Schardin, worked at the Institute on hollow (shaped) charges:

Dr C. Cranz, who published, in collaboration with Dr H. Schardin, the paper in ZPhys **56**, 147(1929) (See pp 6–27 of Porter's)

Prof W. Döring (See pp 28–32 of Porter's)

Prof R. Becker (See pp 36–38 of Porter's)

Prof W. Fuchs, in collaboration with Dr H. Schardin (pp 40–42 of Porter's)]

15) L.H. Eriksen, PATR **1506**(1945) [Properties of Ternary Mixtures of High Explosives (Exudation Study of PTX–1)]

15a) L.H. Eriksen, PATR **1623**(1946) (Properties of Ternary Mixtures of High Explosives. Effect of Storage on Tegnary High Explosives)

16) J. Taylor & S.H. Davidson, USP 2395353 (1946) & CA **40**, 3606(1946) (A TNT expl of d 1.55–1.60 which may be press-loaded is prepd in an Atlas mixer – a jelly of 17 parts NG and 1 p NC with 1 p kieselguhr, 1 p β-naphthyl methyl ether and 80 parts of pulverized TNT. The mixture had to be loaded at once because it sets on standing to a solid mass)
17) W.W. Vogl, USP 2406572 and 2406573 (1946) & CA **41**, 286(1947) (Explosives suitable for loading shells, boosters, etc contn Ethylenediamine Diperchlorate with other ingredients, such as TNT; also an addn compd of Ethylenediamine Perchlorate with PA, etc)
18) V.H. Williams & H.R. Wright, USP 2407595(1946) & CA **41**, 286(1947) [Powerful detonating expls composed of cryst polynitric esters (such as PETN or HN-Mannitol), a nitroaromatic compd liquid at RT and NC with N content >11.8%]
19) H.R. Wright & J. Taylor, USP 2407597 (1946) & CA **41**, 286(1947) (Powerful expl of low sensitivity to impact or friction composed of a mixt of liq Nitrotoluenes, TNT, Tetryl, NC, inorganic nitrates and metal powders)
20) J. Taylor et al, BritP's 578370 & 578371 (1946); CA **41**, 1842(1947) [Incorporation of highly absorbent kieselguhr (5 to 7.5%) into molten TNT produces a cast charge with higher deton velocity than that of cast TNT alone. Other ingredients, such as NG with sol NC may be added. E.g. mixture of TNT 70–85, kieselguhr 5, NG 15–20, sol NC up to 7.5 & stabilizer 5% had density ca 1.6, deton vel up to 6500m/sec and could be initiated by No 6 detonator]
21) H.H. Fassnacht & C.A. Woodbury, USP 2425310(1947) & CA **41**, 6724(1947) (A HE suitable for military and industrial purposes consists of Tetryl 7–90, a liquid aliphatic nitric ester, or an aromatic nitrocompd 5–85 and NC 0.1–7.1%. It is insensitive to rifle bullet impact and possesses high velocity of detonation even after prolonged storage)
22) R.W. Cairns, USP 2430274(1947) & CA **42**, 764(1948) (An explosive suitable for cast loading was prepd by blending 8.5 parts of NStarch moistened with 1.5p alcohol with 10p of pulverized TNT. After drying at RT the mixt is heated at 80–95° to obtain a homogeneous viscous liquid and then poured into molds or shells)
23) V.L. Bobolev, DoklAkadNauk **57**, 789–92 (1947) & CA **46**, 4797(1952) (Limiting diameters of charges of chemically uniform explosives)
24) Arthur D. Little, Inc, "Report on Study of Pure Explosive Compounds", Cambridge Mass: Part 1(1947); Part 2(1947), Part 3 (1950), Part 4(1952)
25) G. Birkhoff et al, JAppliedPhys **19**, 563–82(1948) & CA **42**, 8475(1948) (Explosives with lined cavities – mathematical theory of lining with metal the "Munroe effect" cavity to increase armor piercing capability)
26) G.C. Hale, USP 2472105(1949) & CA **43**, 6827(1949) [Explosives more powerful than TNT and suitable for cast-loading shells, bombs, etc consist of EDNA (Ethylenedinitramine)<60 and TNT >40%]
27) L.F. Audrieth & D.D. Sager, USP 2482089, 2482090 and 2482091(1949); CA **44**, 840(1950) [Three-component expls, such as TNT, Tetryl & RDX; TNT, EDNA & Tetryl or TNT, EDNA & PETN are of high density and are more powerful than TNT. They are suitable for loading, preferably by casting of shells, bombs and mines)
27a) Arthur D. Little, "Synthesis and Testing of High Explosives", Cambridge, Mass, 1st Report (1949), 2nd Report (1951), 3rd Report (1956)
28) F.M. Lang, MP **35**, 213–22(1953) (Solubilities of explosives in several solvents) Translated by N. Gelber, FREL, PicArsn, Translation No 52, Sept 1959
29) A. Schmidt, Explosivstoffe **1954**, pp 121–24, "Die Konstitution reiner explosiver Verbindungen"
30) L. Médard, MAF **28**, 415–92(1954)(169 refs) (Thermochemical properties of explosives. A Table)
31) K. Yamasue et al, JapP 2097(1954) & CA **49**, 2076(1955) (Expls mixts of nitrated naphthalene and toluene with NC, NG, etc)

31a) B.T. Fedoroff et al, "Dictionary of Russian Ammunition and Weapons ", PATR **2145**(1955) (Includes explosives and propellants used by the Russians during WWI and WWII)(Varityped)
32) H. Rosenwasser, USAEC **ORNL-1720**, 28pp (1955) & CA **50**, 14229(1956) (Effects of gamma radiation on explosives)
33) W.S. Cramer, "Bulk Compressibility Data on Several High Explosives", NOLRept NavOrd **4380**(1956)
34) B.J. Zlotucha & M. Baer, PATR **2288** (1956) (Reactivity of explosives with metals and protective finishes)
35) M.A. Cook et al, JACS **79**, 32(1957) (Velocity-diameter curves, velocity transients and reaction rates in PETN, RDX, EDNA & Tetryl)
36) M.A. Cook et al, JPhysChem **61**, 180–96 (1957) (Aluminized explosives)
37) C.G. Dunkle, "Detonation Phenomena", Picatinny Arsenal-Stevens Graduate School Program, Twenty-six Syllabi (1957–1958), Metallized Explosives, p 274
38) P.B. Tweed, FREL, EDS Rept No **23**, March 1958 (The Significance of Explosives in Threads)
38a) B.T. Fedoroff et al, "Dictionary of Explosives, Ammunition and Weapons" (German Section), PATR **2510**(1958)(Varityped 345 double column pages)
39) Poudreries Réunies de Belgique, Rept LC No **186**, October 1958 (Preparation of certain castable explosives based on TNT)
40) J.V.R. Kaufman, ProcRoySoc **A246**, 219–25(1958) (The Effect of Nuclear Radiation on Explosives)
41) E. Piantanida & M. Piazzi, Chim e Ind (Milano) **43**, 1389–93(1961), Engl translation as PATM (PicArsnTechMemorandum) (1962) (Behavior of Explosives Under the Action of Gamma Rays) [Exposure of PA (Picric Acid) and TNT, representatives of the aromatic nitrocompds, to doses as high as 20 megaroentgens for 660 hours did not show any significant decrease in stability as shown by the Taliani Test at 120°C and lowering of the mp. PETN, representative of the nitrate explosives, was reduced rapidly in chemical stability by as much as 0.5 megaroentgens. Nitramine explosives, exemplified by RDX, while not showing the extremely high resistance to gamma radiation as that of the nitroaromatics, resisted satisfactorily 20 megaroentgens. Long exposures to low intensities, 10 roentgens per min for 70 days, cumulative dose of 1 megaroentgen, had very little effect upon the explosives except PETN. However, the effect was very small. Extremely small intensities for very brief periods, (10γ for 0.3 min; 10000γ for 20 min), followed by long aging, showed the radiation had no effect upon the stability of the expls]
42) W.Sh. Dutton, "One Thousand Years of Explosives, From Wildfire to the H-Bomb", Winston, Philadelphia (1960)
43) F. Spring & Ch. Glauser, GerP 1078918 (1960) & CA **55**, 17007(1961) [Desensitized RDX explosive was prepd by kneading a mixt of 69g RDX & 29g Al powder (of 100μ particle size) with 16g of lacquer added by portions. The lacquer consisted of Et cellulose 7.7, di-Bu phthalate 3.3, Centralite 1.3 and various aromatic hydrocarbons 87.7%. Then the mixt was rubbed thru a screen, dried and sieved to obtain 0.5–2.0mm particles]
44) W.A. Gey, USP 2982639(1961) & CA **55**, 17008(1961) [Non-sticky expl compn having low shock sensitivity and high density is obtd by incorporation into HE (such as RDX) ca 10% of an ether of linear Methylenenitramines or mixts thereof having the general structure $RCH_2[N(NO_2)CH_2]_xR'$, where R is H, alkyl or alkoxy; R' is alkoxy and x is an integer. A detailed description of prepn of expl compn consisting of RDX, Di-n-amyl ether of ATX (1,7-Dinitroxy-2,4,6-trinitro-2,4,6-triazaheptane) and "Span" is given]
45) Th.G. Blake, USP 2982640(1961) & CA **55**, 17007 (1961) [Deflagrating expls for loading 20-mm and 40-mm projectiles is provided by a mixt contg 30–70% of finely divided metal (such as Al, Mg, Zn, Zr, Tl, B or alloys), 18–35% of an oxidizer (such as a nitrate or perchlorate of Amm, alkali- or alk-earth metals); 2–4% of TNT (coated on an oxidizer) and 6–30% RDX. Incorporation of 2 parts Cu stearate and 1 p graphite aided in pelleting]
46) J.W. Dawson & F.H. Westheimer, USP 2982641(1961) & CA **55**, 17008(1961) [Alumized

expls with greatly reduced or eliminated spewing and gas-generating tendencies were obtd by incorporating a hydrophilic or desiccating agent, such as $Mg(NO_3)_2$, $CaCl_2$, or $Mg(ClO_4)_2$. This compn contg Al (fine) 18, RDX 42 & TNT 40%, with added 0.5% of fine anhyd $CaCl_2$ had a sharply reduced tendency to spew and generate gas both while pouring and after introduction into containers]
47) P. Aubertein, MP **43**, 39–62(1961) (Studies of phlegmatized expls)
48) Armour Research Foundation, "Experimental Studies of the Effects of Impact Loading on Plastic-Bonded Explosive Materials", Final Rept, 1 Oct 1961 to 20 Feb 1963 (Contract DA–29–0440XZ–885)
49) Naval Ordnance Laboratory, "Heat Resistant Explosives XV", NOLTR **62–175** (1962)
50) Aerojet-General Corp, "Investigation of High Explosives at Elevated Temperatures", Final Rept No 0136–02(12)FP, Dec 1962, Contract NOrd 17881
51) M.J. Urizar et al, Explosivstoffe **10**, 55–63 (in Engl) & 64–6 (in Ger) (1962) (Effects of nuclear radiations on organic expls)
52) K.K. Andreev, Explosivstoffe **10**, 203–12 (1962) & CA **58**, 3263(1963) (Thermal decompn and combustion processes of expls)
53) K.K. Andreev, DoklAkadNauk **146**, 413–14 (1962); JApplChem **13**, 487(1963) and Explosivstoffe **11**, 275-M(1963) (Difference between primary and secondary expls)
54) M.F. Murphy, "Two Explosives Generating Condensible Products", NOLTR **63–12**(1963) [Mixts studied were Lithium Perchlorate Trihydrate (3 moles) & Aluminum (8 moles) and amorphous Al powd with aq hydrogen peroxide (90%)]
55) N.L. Coleburn, "Chapman-Jouguet Pressures of Several Pure and Mixed Explosives", NOLTR **64–58** (1964) (Cast chges of TNT, Comp B & Pentolite and pressed RDX, PETN & Tetryl)
56) Anon, Ordn **51**, 94(July–Aug 1966) [New expls **Astrolites** have been produced by the Explosives Corp of America, a subsidiary of Rocket Research Corp. It is claimed that some of these expls are more powerful than Blasting Gelatin. Their compns are not given but it is evident that they are different from those of *Astralites*, described in Vol **1** of Encycl, pp A497-R & A498-L and in PATR **2510**(1958), p Ger 10. See Ref 63a]
57) PATR **2700**, Vol **3**(1966), pp D38-R to D40-L (Deflagrating expls; definition)
58) PATR **2700**, Vol **3**(1966), p D107 (Detonating expls; definition)
59) DuPont's Blasters' Handbook (1966) (Expls of DuPont & Co)
60) Heizo Nambo, "Who Invented the Explosives", Journal of the Industrial Explosives Society of Japan **28**, 322–29 & 403–13(1967). Engl translation in Japanese Studies in the History of Sciences No 9(1970). Obtainable from Japan Publishers Trading Co, Ltd, PO Box 5030, Tokyo International, Tokyo, Japan. Price $15 by surface mail. Briefly reviewed in Expls&Pyrots **5**(11), Nov 1972
61) W.R. Tomlinson Jr & E.E. Sheffield, PATR **1740**, revised and published as Army Materiel Command Pamphlet, AMCP **706–177** (1967 & 1971) under the title "Properties of Explosives of Military Interest", Washington, DC 20315. Available as AD–814964 from Defense Documentation Center, Cameron Center, Alexandria, Virginia, 22314. Briefly reviewed in Expls&Pyrots **1**(9), 1968
62) Anon, "Military Explosives", Department of the Army Technical Manual **TM 9–1300–214** and Department of the Air Force Technical Order **TO 11A–1–34** (Nov 1967), superseding TM 9–1910/TO 11A–1–34(1955), Washington, DC 20315
63) Anon, "Explosives and Demolitions", **FM 5–25**(1967), p 155-R (Sheet explosive M118 for use in military demolition block, weighing 0.90 kg. No compn given but its deton velocity is 7190m/sec)
63a) S. Levmore & S.J. Lowell, "Principal Characteristics of the Liquid Explosive, Astrolite G", PATR **3633**(1967) (A liq expl based on Hydrazine)
64) N.E. Beach & V.K. Canfield, "Compatibility of Explosives with Polymers", Plastec Rept **33**, PicArsn 1968 (AD–673 713) and Plastec Rept **40**(1971) AD–721 004). Also M.C. St Cyr, PATR **2595**(1959) (AD–714 634) and N.E. Beach, "A Guide to the Reactions

Reported in PATR 2595", Plastec Note **22** (1970) (AD–716 624). All obtainable from NTIS, US Dept of Commerce, PO Box 1553, Ravensworth, Va 22151

Reviewed by G. Cohn, Edit in Expls&Pyrots **6**(4), 1973. (This compilation records compatibility testing at Picatinny Arsenal. Compatibility indicator was the result of gas evolution determined in the vacuum stability test. Reports present the information in two ways: by generic name or trade name of the plastic and by explosive. The reader can quickly scan the information to see with what expls a plastic is compaticle and what plastics can be used safely with a particular explosive)

65) Bruce Anderson, Ordn **53**, 613–15 (1969) [The values of so-called *Figure of Merit* (gas volume times heat of explosion) are given for *Astrolite A-1-5* as 1600, while for *Astrolite G* as 1175, in comparison with TNT 675, Tritonal 792 & RDX 1162.

No compns of Astrolites are given]

66) PATR **2700**, Vol **4**(1969), pp D574-R & D575-L (1969) (States properties of explosives)

67) Anon, Atlas/Aerospace No 8, 1969, Atlas Chemical Ind, Valley Forge, Pa 19481; reviewed by G. Cohn, Edit in Expls&Pyrots **3**(3), 1970 under the title *Explosives for Actuators* [LMNR (Lead Mononitroresorcinate), KDNBF (Potassium Dinitrobenzofuroxane) and BaSt (Barium Styphnate) are the three common materials used as either ignition drops or base charges in actuators, motors, switches, and matches. LMNR, a stable, low-cost compd, is always used with 5 to 40% $KClO_3$. KDNBF, a higher cost material, is 5 to 10 times more powerful than LMNR. BaSt is 80% as powerful but a better gas producer than KDNBF. Their Explosion Temperatures (°C at 5 sec) are 255–285° for LMNR, 250° for KDNBF, 250° and 341° for BaSt, while their Impact Sensitivities by 2-kg Drop Weight Test, are 45, 2 and 120cm]

68) C.L. Scott, "The Sensitivity of Selected Conventional and Heat Resistant Explosives at Low Temperatures", NOLTR **70–36** (1970), reviewed by G. Cohn, Edit in Expls&Pyrots **4**(6), 1971 [The effects of temp, confinement, and column diam on the shock sensitivity were studied in a gap test arrangement for the following expls: Tetryl, CH-6, HNS-II, DIPAM (3,3′-Diamino-2,4,6,-2′,4′,6′-hexanitrobiphenyl), TACOT-Z (Tetranitro-1,2,5,6-tetraazadibenzocyclo-octatetrene) and TATB (1,3,5-Triamino-2,4,6-trinitrobenzene). Test temps were ambient and –68°C. As expected, sensitivity decreased with decreasing temp. These changes were small compared to the sensitivity effects of confinement and charge diameter]

69) Anon, "Fuze Catalog", **MIL-HDBK-137**, 20 Feb 1970. [Complete listing of military and technical info for all std and devel US items. Current fuzes (Vol 1), obsolete and terminated fuzes (Vol 2), fuze explosive components (Vol 3)]

70) B.M. Dobratz, "Properties of Chemical Explosives and Explosive Simulants", Lawrence Livermore Laboratory, Univ of Calif Rept **UCRL–51319** (Dec 1972) (AEC Contract W–7405–Eng–48) (A tabulation of names, formulations & props of various expls & energetic compds)

71) **CG108**, "Rules and Regulations for Military Explosives and Hazardous Munitions" (Revised annually), US Coast Guard, 400 7th St NW, Washington, DC 20591 [See Rules and Regulations for Explosives in General under EXPLOSIVES NONMILITARY (COMMERCIAL)]

72) Anon, "Explosive Trains", **AMCP 706–179** (1973)

EXPLOSIVES NONMILITARY (COMMERCIAL) (Papers and Reports Listed in Chronological Order)

In addition to references listed in Vol **3** of Encycl on pp C456-R to C459, under COMMERCIAL OR INDUSTRIAL EXPLOSIVES and in Vol **5** on pp D1649-L to D1722-R, under DYNAMITE, the following may be included:

1) Atlas Powder Co, Fire and Water Eng **74**, 151 et seq (1923) & CA **17**, 3254 (1923) (Application of expls in checking large fires is discussed)

2) Bombrini Parodi-Delfino, FrP 793591 (1936)

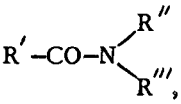
& CA **30**, 4674(1936) [Gelatinous expls are obtained by using PETA (Pentaerythritol tetracetate) in conjunction with NC, TNT or PETN

3) Imperial Chem Inds of Australia & New Zealand, AustralianP 101287(1937) & CA **31**, 7252(1937)(Gelatinous & semigelatinous expls are improved as regards plasticity and safety by adding china clay, bentonite or other clays in amts 2–6%)

4) J.T. Power & K.R. Brown, USP 2102187 (1937) & CA **32**, 1455(1938)(Gelatinization of NC with an organic nitric ester is improved by incorporating a small amt of an accelerator such as ethoxyethyl nitrate or acetate)

5) W.H.W. Williams & B.W. Foster, BritP 460576(1937) & CA **31**, 5583(1937); USP 2129367(1938) & CA **32**, 8782(1938)(Same as AustralianP 101287, listed as Ref 3)

6) W.E. Lawson, USP 2159973(1939) & CA **33**, 7115–16(1939) [For accelerating gelatinization of expls such as those contg NC & NG or NGc, a small quantity (0.5–2%) of dimethyl formamide is incorporated. Other acid amides of the general formula



, in which R′, R″ & R‴ stand for H, or alkyl radicals]

7) H.J. Tettersall, USP 2188322(1940) & CA **34**, 3918(1940)(A Pb salt of dimethylphosphoric acid is used in gelatinized NC compns to reduce their inflammability)

8) O.J. Otto, ExplosivesEngr **17**, 376(1939) & CA **34**, 887(1940)(When an oil-hole drill broke off in the crankshaft, 1/3 to 1/2 thimbleful of 40% ammonia gelatin dynamite was pushed down the hole and a cap and fuse were placed on the dynamite. After firing the charge, the drill piece fell out on tapping with a hammer)

9) D.G. Morrow, CanadP 414716(1943); CA **37**, 6462(1943)(A mixt of lower paraffins, such as nitromethane in nitrobutane 35–90% and liquid N_2O_4 65–10%)

10) M.F. Lindsley Jr, USP 2320971 and 2320972(1943) & CA **37**, 6896(Explosives based on granular AN)

11) R.F.B. Cox, USP 2330112(1943) & CA **38**, 1368(1944) (Explosive contg Nitroisobutanol Nitrate, AN and other ingredients)

12) K.E. Riper, USP 2334149(1943) & CA **38**, 2824(1944) (Explosives consisting of NG held within the pores of porous condensation product of urea and formaldehyde)

12a) F.R. Benson, PATR **1284** & **1287**(1943); **1338**(1943) & **1418**(1944)(Explosive scrap recovery)

13) R.W. Lawrence, CanadP 417844(1944) & CA **38**, 1644(1944) (Expl contg concd nitric acid and nitroparaffin, such as nitric acid 15–63%, nitromethane 85–37%)

14) C.H. Carey, USP 2345582(1944) & CA **38**, 4447(1944) (An expl contg at least 60% AN, 5–12% of fatty materials, such as petrolatum and 5–25% carbonaceous material, such as starch, cereal flour etc and other ingredient and prepd by a special method)

15) M.A. Cook & C.O. Davis, USP 2353147 (1944) & CA **38**, 6098(1944) (Explosive of high density is prepd by heating a mixt of TNT, AN and a dispersing agent such as Na alkyl naphthalene sulfonate to a temp at which at least a portion of TNT is in molten condition)

16) C.O. Davis, USP 2358384(1944) & CA **39**, 1760(1945) (Detonating explosives, such as AN 74.5, TNT 10.0, sulfur 2.0, woodpulp 6.0, a flaked corn product 7.0 and chalk 0.5%. Other mixtures may be used)

17) K.M. Gaver, USP 2389771(1945) & CA **40**, 1036(1946) (Low density expl contg starchate of alkali metal or ammonia in which the metal is attached through an O atom to a C in the 2 position in the starch mol. Thiochloro, thioammonium or nitrostarches may also be used. E.g: NH_4 starchate, thiochloro, thioammonium 75 & NG 25%)

18) J. Whetstone, USP 2409919(1946) & CA **41**, 865(1947) [Low density expls are prepd by emulsifying easily melting expl (such as TNT) by a colloid (such as agar-agar or Na alginate) in a satd NH_4NO_3 soln contg solid AN in suspension (expl with higher mp such as PETN may be mixed with TNT). The mixt is congealed, granulated and dried at low temp]

19) H.R. Wright et al, BritP 577896(1946) & CA **41**, 1841(1947) (Plastic explosive con-

sisting of a polynitrate ester, a nitroaromatic and NC)
20) J. Taylor et al, BritP 578371 (1946) & CA **41**, 1842 (1947) (Gelatinous expl suitable for demolition purposes and as bursting chge for bombs, shells, etc is prepd by mixing a jelled NG with TNT, stabilizer, etc)
21) J. Taylor et al, BritP 579376 (1946) & CA **41**, 1842 (1947) (Plastic explosives consisting of a liquid nitrate ester and a 1:3 mixture of sol:insol Nitrocelluloses)
22) P.G.R. Bacon et al, BritP 580418 (1946) & CA **41**, 1841 (1947) (Plastic expls consisting of 1:1 PETN:TNT plasticized with PVC & dibutyl phthalate)
23) C.R.S. Harries et al, BritP 588885 (1946) & CA **41**, 1842 (1947) (Chopped and shredded straw is incorporated in gelatin expls, such as those contg NG & NC)
24) A.Ya. Apin & V.K. Bobolev, ZhFizKhim **20**, 1367–71 (1946) & CA **41**, 3297 (1947) (The effect of physical structure and state of aggregation on the sensitivity of expls) (Engl translation OTIA **5377**, AEC–tr–3781)
25) D. Harrington & J.H. East Jr, USBurMines InfoCirc **7380**, 30pp (1946) & CA **41**, 4309 (1947) (Handling, storage and use of expls in mines and quarries)
26) P.W. Bridgman, "The Effect of High Mechanical Stress on Certain Solid Explosives", JChemPhys **15**, 311–13 (1947)
27) G. Herzberg & G.R. Walker, Nature **161**, 647–48 (1948) (Initiation of HE's)
28) J. Taylor, Research (London) **1947**, 67–76 & CA **42**, 2105 (1948) (Expls; review with 59 refs)
29) A.F. Belyaev, ZhurFizKhim **22**, 91–101 (1948) & CA **42**, 5227 (1948) (Boiling points, heats of vaporization and Trouton constants of secondary expls)
30) J. Weir, Nature **158**, 83–5 (1946) & CA **43**, 2435 (1949) (History of expls)
31) R. Saint-Guilhem, RevIndMinérale No **551**, 791–810 (1949), No **552**, 3–23 (1950) & CA **44**, 5099 (1950) (Mining expls)
32) E.H. Buchanan, "Explosive Screw", PATR **1769** (1950) (See description earlier in this Volume under **Explosive Screw**)
33) R. Leclercq, IndChimBelge **15**, 3–10 (1950) & CA **44**, 5594 (1950) (Expls; review with 34 refs)
34) K.K. Andreev & M.A. Rabinovich, ZhurFizKhim **24**, 650–61 (1950) & CA **44**, 11097 (1950) (Mining explosives)
35) M.M. Thacker, H.O. Folkins, USP 2460200 (1949) & CA **45**, 10565 (1951) (Addition of small quantities of explosives, such as Ethyl Hypochlorite, TNT & Benzazide etc to the hydrocarbons undergoing cracking, increases the yields of desirable products, permits carrying the reaction at lower temp and in shorter time)
36) L.W. Braniff, WorldOil **131**, No 7, 73–5, 90 (1950) & CA **45**, 1345 (1951) (Expls for seisomographic prospecting for oil)
37) J. Tanaka, Reports of Osaka Prefecture IndResearchInst **2**, 27–33 (1950); Ibid **3**, No 3, 5–12 (1951); Ibid **4**, No 1, 32–6 (1952) & CA **46**, 11743 (1952) (Explosives Used in Manuf of Cellular Rubber) {Some primary expls, such as Diazoaminobenzene or Azides (of Ca, Ba or Na) had been used in Japan as cellulating agents in manuf of cellular rubber. For example, 100 parts of crude rubber was thoroughly mixed with 6ps of Diazoaminobenzene, $C_6H_5.N:N.NH.C_6H_5$, 6ps stearic acid, 3ps ZnO, 3ps sulfur and 3ps tetramethylthiuram disulfide, $[(CH_3)_2NCS]_2S_2$, and the resulting mixture was vulcanized by heating 10 mins at 115°, twice in succession in a press capable of exerting 20kg/sq cm pressure}
38) J.M. Downard, USP 2537039 (1951) & CA **45**, 2670 (1951) [Gelatinous expls of low or zero NG content are prepd by adding 2–3% of a non-expl gel, such as prepd by mixing 95% naphthene base oil (η_{25} = 1000–2000 centipoises) with 5% of polyisobutylene as a gelling agent]
39) B. Kubalov, VzryvnoeDelo **1949**, No 43 & CA **46**, 5843 (1952) (History of expl in USSR in mining and construction)
40) H.C. Grimshaw, Ministry of Fuel and Power, Safety in Mines Res Estab, Res Rept No **34**, 3–29 (1951) & CA **46**, 6385–86 (1952) (Slow burning of permitted expls as a possible cause of hangfires or delayed explns)
41) H. Henkin & R. McGill, IEC **44**, 1391–95 (1952) & CA **46**, 8857 (1952) (Rates of expl decompn of expls. Exptl & theoretical

kinetic study as a function of temp)
42) A. LeRoux, RevIndustrMinérale **33**, 276–84(1952) & CA **46**, 10623(1952) (History of development of explosives industry)
43) P. Tavernier, MP **32**, 239–53(1950) & CA **47**, 9014(1953) (History of smokeless powder explosives)
44) E. vonHerz Sr & Jr, GerP 859868(1952) & CA **52**, 14171(1958) (Decrease in sensitivity of gelatinous expls due to prolonged storage can be prevented by incorporating expls which are insol or only partly sol in expl oils. TNA and its derivs are the most effective. Eg: a) NGc 28.5, NC 1.5 & TNA 70% – very brisant expl and b) NGc 47.5, NC 2.5, TNA 10 & AN 40% – medium brisant expl)
45) G.B. Kistiakowsky et al, USP 2606109 (1952) & CA **47**, 4083(1953) (Plastic non-hardening expl prepd from 88% Cyclonite and 12% bodied or polymerized oil which may be distilled from sperm oil, rape seed or petrolatum)
46) L. Médard, MP **32**, 209–25(1950) & CA **47**, 9013(1953) (Mining expls in France, review)
47) J. Taylor, AnnReptsSocChemIndProgress-ApplChem **35**, 290–315(1950) & CA **48**, 13128(1954) (Explosives; review of 1939–1950)
48) J. Taylor & E. Whitworth, AnnReptsSoc-ChemIndProgressApplChem **37**, 524–38(1952) & CA **48**, 13130(1954) (Explosives; review)
49) R.C. Clogau, USP 2676878(1954) & CA **48**, 13223–24(1954) (Gelatinized high expl compns prepd by incorporating in a blend of NG, NGc, NC, DNT, AN, K nitrate, carbonaceous material & sulfur, a small amt of a salt of triethanolamine and a higher fatty acid blended with at least one monohydric aliphatic alc contg 10–18 C atoms in the chain)
50) H. Zenftman & E. Whitworth, BritP 713758(1954) & CA **49**, 3537(1955); USP 2716056(1955) & CA **49**, 15242(1955) [Power to quench the expln flame is increased by incorporating some cryst salts in gelatinous expls based on NG–NC gel, AN, woodmeal, etc. In an example, 0.20 part p-tert-octylphenyldiethylphosphate and 3 parts o-MNT are dissolved in NG+NGc in the proportion 80:20 and to this NC (1.1 parts) is added. The resulting gel is mixed with other ingredients]
51) A. Stettbacher, "Sprengstoffe und ihre Wirkung in Theorie und Praxis" (Explosives and Their Effect in Theory and Practice), Explosivstoffe, Vol **2**, 39–41, 113–21 & 149–57(1954); Vol **3**, 9–11, 41–6, 118–21, 157–60 & 178–82(1955)
52) H. Selle, Explosivstoffe **3**, 114–16(1955) (Risks of ignition accompanying the breakage of low pressure vessels)
53) US Interstate Commerce Commission, "Transportation of Explosives and Other Dangerous Articles by Express, Including Specifications for Shipping Containers", Railway Express Agency, NY(1955), Suppl 1, 5
54) L. Médard, MAF **29**, 669–88(1955) (Safety of explosives)
55) E. Whitworth, AnnReptsSocChemInd-ProgressApplChem **39**, 305–22(1954) & CA **50**, 509(1956) (Explosives; review with 268 refs)
56) A. Cachin, IndChimBelge **20**, Spec No, 217–20(1955) & CA **50**, 6796(1956) (Some permissible expls of the reduced density type)
57) B.P. Enoksson, USP 2736742(1956) & CA **50**, 6796(1956) (Preparation of some nitrated organic products)
58) P.G. Wallerius, SwedP 152025(1955) & CA **50**, 7463(1956) (Plastic expls from liquid organic nitrocompds, Al powder and inorg nitrates)
59) K. Namba, JIndExplSocJapan **16**, 70–9, 132–43(1955) & CA **50**, 17451(1956) (Explosives; review with 185 refs)
60) M.A. Cook & M.T. Abbegg, IEC **48**, 1090–95(1956) (Isothermal decompn of expls)
61) A. Langhans, Explosivstoffe **4**, 15, 53, 79, 148, 177 & 230(1956) and Ibid **5**, 12 & 33 (1957) (Explosives; Applications of Non-explosive Nature)
62) Kuhn & Käufer, Explosivstoffe **4**, 267 (1956) (The historical comprehension of the practical work and the mechanism of the behavior of explosives)
63) L. Médard, "Physics of Explosives",

MAF **31**, 553–55, 556–69 & 570–83(1957); Ibid **32**, 911–52(1958) and Ibid **33**, 7–30 & 31–58(1959)
64) WASAG Chemie AG, GerP 1036137(1958) & CA **54**, 6833(1960) (Expls of high mechanical strength prepd by working into expls, as carrier structures, organic fibrous substances, especially nitrated ones)
65) H.W. Koch & H. Freiwald, Explosivstoffe **6**, 279–84(1958) (Pourable expls and determination of their viscosity)
66) H.P. Tardif, Materials in Design Engineering **49**, No 2, 82–7(1959) (A roundup of present applications of explosives to form, fasten, engrave & test)
67) P. Aubertein, MP **41**, 111–25(1959) (Stability of expls)
68) G. Boyd & A.C.P. McIntosh, AustralP 213095(1958) & CA **54**, 16833(1960) (Water-resistant expls contain 0.2–6% lanolin)
69) K.K. Andreev, Explosivstoffe **8**, 102–11 (1960) & CA **54**, 23330–31(1960) (Thermal decompn & burning of expls)
70) N. Griffiths & J. Groocock, JCS **1960**, 4154–62 and Explosivstoffe **11**, 249(1963) (Burning to detonation of solid expls)
71) L. Deffet, Explosifs **13**, 103(1960) and Explosivstoffe **12**, 195(1964) (Development of expls in Belgium)
72) R.W. VanDolah, IEC **53**, No 7, pp 59A to 62A (Intern Ed 50A–53A)(1961) & CA **56**, 619(1962) and Explosivstoffe **10**, 249(1963) (Evaluating the expl character of chemicals) (Available testing procedures prove to be inadequate for reliable results; difficult to test for the "unexpected" things people do with chemicals)
73) F.C. Gibson et al, "Sensitivity Characteristics of Liquid Explosive Systems", US-BurMinesProgrRept No **3**, July 1 to Sept 30 1962, Contract No 19–62–8021–WEPS
74) N.S. Bakharevich et al, Vzryvnoe Delo, NauchnoTekhnGornoyeObshchestvo, Sbornik **1962**(49/6), 190–201 & CA **59**, 3710–11 (1963) (Permissible expls for mines with hazardous sulfide dust) (A mixture of w-resistant AN, TNT, sawdust, ammonium chloride flame quencher and 0 or 9% liquid nitroester is permissible for use in a copper pyrite or similar mine with sulfide dust. With 9% nitroester the formulation (designated VS-1) is suitable for 1.25–1.40 inch cartridges, while up to 3.9 inch cartridges may be used with 0% nitroester (VS-2). For VS-1 and -2, respectively, the following props are found: heat of expln (kcal/kg) 742.16 and 718, brisance (mm) 17.26 and 14.5, distance of deton transmission (cm) 11 and 3.8, max charge for sulfide dust (gm) 450–600 and , expansion in a lead bomb (ml) 280 and 260)
75) Inspection Générale des Poudre & C. Napoly, "Accidents at Explosives Plants", MP **45**, 224–77(1963) (Description of 69 accidents which took place at various French expls plants during 1956–1960)
76) CanadIndsLtd, CanP 658221 and Kemixon Reporter, October 1963, p 1168, Item 10–614–3 [Explosives in suspension such as AN 37.5, $NaNO_3$ 24.5, TNT(in pieces) 25.0 & carbon black 1.0 suspended in 12.0 parts water]
77) G.F. vonKrogh, Explosivstoffe **11**, 275-L (1963) (Inexpensive expls with addn of water) (Prepd from AN, sodium nitrate, sodium chlorate, TNT, and thickened with guar gum, sodium CMC or sodium alginate)
78) F. Müller, Explosivstoffe **11**, 269-R(1963) "Explosives in Powder Form With Reduced Sensitivity to Impact" [Addn of 0.1% of 4-aminodiphenylaminosulfonic acid to composition consisting of AN 80, NG 6, TNT 12 & woodmeal 2% changed its sensitivity to impact from 1.0mkg to 2.5mkg. In case of expl contg AN 82, TNT 11, Al 4 & woodmeal 3%, the addn of 0.1% of the above compd chan ged its sensitivity to impact from 1.0 to 1.5mkg. The gap test value (Übertragungswerte) was not changed in the case of the 1st expl and increased only slightly in case of the 2nd expl (from 10cm to 11cm)]
Note: Accdg to Kemixon Reporter, Nov 1963, p 1312, Item 10–6189, the following compn was of Wasag-Chemi, GerP 1143425: AN 82, TNT 15, DNT 1 & woodflour 2% with added 0.05% of desensitizer, 4-amino-diphenyl amino-sulfonic acid or 4,4′-diamino-diphenyl amino-sulfonic acid had impact sensitivity value using Koenen app 2.0m/kg, vs 0.75m/kg for the same expl without added desensitizer

79) V.S. Ilyukhin & P.F. Pokhil, "The Sensitivity of Some Explosives to a Shock Wave", DoklAkadNauk, (1961), English Translation from Russian by W. Urusky, Redstone Scientific Info Center **RSIC-122** (Jan 1964)
80) F.C. Gibson et al, "Sensitivity Characteristics of Liquid Explosive Systems", BurMines ProgrRept No **10**, 1 Apr-30 June, 1964, Contract IPR-19-64-8026-WEPS
81) Poudrerie Royale de Wetteren SA, BelgP 654268(1965) & CA **64**, 11022(1966) (Flexible expls are prepd from Nitroglycerin and/or Nitroglycol with plasticized NC and red lead stabilizer. More flexibility can be had by adding dibutylphthalate)
82) PATR **2700**, Vol **3**(1966), p C500 (Note with 2 refs) (Construction of Buildings and Installations for Explosives and Propellant Plants)
83) S.B. Wright, "Granulated Crystalline Plastic Bonded Explosives", USP 3396041 (1967) & CA **66**, 87231-R(1967). Continuation in part of USP 3173817(1965), listed in CA **62**, 12968(1965) and in USP 3138501(1964), listed in CA **61**, 10530(1964)
84) H.H. Mohaupt, "Pelleted Ammonium Nitrate Blasting Explosives", USP 3301722 (1967) & CA **66**, 87228(1967) (AN 68, TNT 30, paraffin wax 2%, mixed and extruded under high pressure to give sp gr 1.4 pellets ¼-inch diam)
85) Anon, "Active List of Permissible Explosives and Blasting Agents Approved Before December 1, 1967", BurMines Information Circular **8371**, April 1968, US Dept of the Interior, 4800 Forbes Ave, Pittsburgh, Pa 15213. Reviewed by G. Cohn, Edit in Expls&Pyrots **1**(6), 1968
86) P.A. Richardson & C.M. Mason, "Active List of Permissible Explosives and Blasting Devices Approved Before July 1, 1970, BurMines Information Circular **8493**, Oct 1970. Reviewed by G. Cohn, Edit in Expls&Pyrots **4**(1), 1971 .(The list of permissible explosives includes 76 brands of which 11 are gelatinous. The list of permissible blasting devices comprises five Cordox models)
87) Gunther Cohn, Edit, Expls&Pyrots **4**(8), 1971, "ON SHIPPING EXPLOSIVES" and in Vol **5**(1), 1972, "MORE ON SHIPPING EXPLOSIVES". Shipping regulations are complex and a qualified shipper is needed to cope with them. The shipper must be properly licensed in all countries and in each US state involved. All cargo of hazardous material must be properly blocked and braced during shipment. For some hazard classes, the vehicle must be placarded and inspected. Mixed shipments in the same vehicle must be compatible. Whether shipment is planned by rail, truck, ship or air, it is desirable to follow the regulations for all modes of travel to avoid problems in case it is necessary to change the shipping mode

All of the refs below are updated periodically, most of them annually. Refs A-F are from Expls & Pyrots, G is an update of A, and H is an addnl ref. More detailed information can be obtd from Mr William Byrd, Deputy Director, Dept of Transportation, Office of Hazardous Materials, 400 6th St, SW, Washington, DC 20590
Refs: A) Tariff 23, "Regulations for Transportation of Explosives and Other Dangerous Articles by Rail", published by Agent T.C. George, 2 Penn Plaza, New York, NY 10001 B) Tariff 11, "Regulation for Transportation of Explosives and other Dangerous Articles by Truck", published by Agent F.G. Freund, American Trucking Assoc, Inc, 1616 "P" St, NW, Washington, DC, 20036 C) CG108, "Rules and Regulations for Transportation of Military Rxplosives and Hazardous Munitions by Ship", US Coast Guard, 400 7th St, NW, Washington, DC 20591 D) Tariff 6-D, "Official Air Transport Restricted Articles Tariff", published by C.C. Squire, Airline Tariff Publishers, Inc, Agent, 1825 "K" St, NW, Washington, DC 20006 E) Code of Federal Regulations, **Title 49**, Transportation, Parts 170 to 179. Available from the Superintendent of Documents, US Govt Printing Office, Washington, DC 20402 F) **Title 46**, Parts 146-149 covers shipping by water and **Title 14**, Part 103, covers shipping by air G) Tariff 25, "Hazardous Materials Regulations of the Department of Transportation", published by Agent R.M.

Graziano, Assocn of American Railroads, Washington, DC H) **OP 2165**, "Navy Transportation Safety Handbook''

88) R.A. Dick, "Current and Future Trends in Explosives and Blasting". Pit and Quarry **64**, Pt 1, No 1, July 1971, pp 159–62 & 174; Pt 2, No 2, Aug 1971, pp 105–07; reviewed by G. Cohn, Edit, in Expls&Pyrots **4**(12), 1971. [This paper is an attempt to analyze current trends in the expl field and to project developments that are likely to occur in the near future. During the past 15 years, while inflation was steadily increasing, the costs of most phases of blasting operations in mining and construction work were steadily decreasing. This improvement in costs can be attributed to: 1) The continuing development of lower cost and more efficient blasting expls (such as ANFO and slurries); 2) The presently depressed price of AN; and 3) More inexpensive and efficient methods of loading these products

In future, continued reduction in blasting costs will be realized thru improved formulations of existing product types and more widespread use of bulk loading techniques. Dry blasting agents such as ANFO will continue their dominance in terms of product usage, while slurries will gradually increase their share of the market. Expl-based slurries will largely be replaced by aluminized or other type slurries. Cap-sensitive slurries with small critical diams will replace Dynamites in many cases, and lower-energy, low-priced slurry products will be developed to compete with ANFO in some large-diam applications. Pneumatic loading of ANFO will find increased applications in open cuts and in underground mining, while bulk slurry loading, already widely used in large operations, will find increased application in small-diam work]

89) PATR **2700**, Vol **5**(1972), "Explosives of Apache Powder Co", Benson, Arizona 85602, are listed under DYNAMITE, Tables IV, VII, VIII, IX and X

89a) PATR **2700**, Vol **5**(1972), "Explosives of Trojan Powder Co", now known as "Trojan-US Powder", Division of Commercial Solvents Corp, 17 North 7th St, Allentown, Pa 18105, are listed under DYNAMITE, Class VIII & X

90) R.J. Buxton & T.M. Massis, "Compatibility of Explosives with Structural Materials of Interest", Sandia Laboratories", Aug 1970 & June 1972; reviewed by G. Cohn, Edit, in Expls&Pyrots **6**(2), 1973 (Intended as an aid to the design engineer, the reports list 1500 structural material combinations and label them as compatible, incompatible, or marginal. The structural materials include: metals, plastics and adhesives, while the expls include: primary and secondary expls, propellants and pyrotechnics)

91) C.M. Mason & E.C. Aiken, "Methods for Evaluating Explosives and Hazardous Materials", BurMines Information Circular **8541** (1972). Reviewed in Expls&Pyrots **5**(9), 1972 (The methods currently used by the Bureau of Mines to evaluate the sensitivity, strength, incendivity, gaseous products, and physical properties of explosives and hazardous chemicals are described. Included are the tests used to evaluate explosives which are certified as permissible for use in underground coal mines)

92) Capt H.H. Reed, "Slurry Explosives", Army Res & Devel News Magazine, Vol **13**, No 8, pp 22–3, Dec 1972. Reviewed by G. Cohn, Edit, in Expls&Pyrots **6**(5), 1973

SLURRY EXPLOSIVES. To comprehend fully the evolution of slurry explosives, one must start with the discovery that by adding fuel oil to pelletized ammonium nitrate, an excellent explosive for production blasts was obtained. As ammonium nitrate was a relatively inexpensive fertilizer, much safer and easier to work with than cartridged dynamites, it soon gained wide acceptance in the drilling and blasting industry. The ammonium nitrate currently used in blasting is formed into a porous pellet called a prill. A mixture of 94.5% AN and 5.5% fuel oil was found to be the most efficient explosive and is termed ANFO. The addition of fuel oil allows all the available oxygen from the ammonium nitrate to be used effectively in the explosion

Unfortunately, ANFO is not without se-

Measured Properties and Calculated Parameters Of Representative Cratering Explosives

Explosive	Detonation pressure (kbar)	Bulk specific gravity	Detonation velocity (m/sec)	Impedance (m/sec)	Heat of detonation (cal/g)	Nominal cost ($/lb)	Excavated volume relative to equal weight of TNT[a]
ANFO	60	0.93	4560	4240	890	0.06 ± 0.04	1.0 - 1.1
AN Slurry	104	1.40	6050	8470	730	0.15 ± 0.05	1.0 - 1.2
AN Slurry (2% Al)[b]	60	1.30	4300	5590	750	0.08 ± 0.05	1.0 - 1.2
AN Slurry (8% Al)[b]	66	1.33	4500	5990	1110	0.13 ± 0.05	1.2 - 1.4
AN Slurry (20% Al)[b]	85	1.20	5700	6840	1450	0.20 ± 0.07	1.5 - 1.7
AN Slurry (35% Al)[b]	81	1.50	5000	7500	1950	0.25 ± 0.10	1.6 - 1.8
TNT	220	1.64	6930	11360	1102	0.25 ± 0.05	1.00

[a] *That is, "Cratering Effectiveness" as measured by small charge detonations in sand. Absolute cratering performance in terms of volume excavated per pound of explosive depends on the size of the shot; it is less for larger shots. Relative performance, on the other hand, is not as sensitive to charge size.*

[b] *Slurry blasting agent.*

veral drawbacks, including its low density and its hygroscopicity (ability to absorb moisture). Paradoxically, it was found that thru the addition of water, with suitable stabilizing agents, and, if desired, gelling agents, not only are most of these problems overcome, but handling of the explosive is simplified. These water-based slurries or gels vary in consistency from a heavy paste or jelly to a solid rubbery mass, depending on the gelling agents used. The most common agent is a gum, such as guar gum. The gelling agent serves two purposes: (1) it insures a homogeneous mixture by preventing settling of components, and (2) it facilitates handling

A simple slurry is one in which the fuel oil found in ANFO is replaced with another fuel which is compatible with a water gel. Most commercial slurries consist of an explosive base, such as ammonium nitrate or sodium nitrate, and a fuel, such as carbon, sulfur or aluminum. The addition of large quantities of alluminum produces a slurry with very high energy release at moderate detonation pressures

The table indicates that both ANFO and slurries are excellent cratering explosives the effectiveness of which equals or exceeds TNT capabilities. Advantages are ease of field use, insensitivity to heat and shock – hence safe, voidless filling of holes, and low expense. Disadvantage is the need for a large booster charge for initiation

93) T. Ito, KogyoKayaku **31**(3), 124–30 (1970) & CA **78**, 6067 (1973) (Deflagration of Ammonium Perchlorate) (Review with 49 refs dealing with controlled experiments)
94) O. Katsuki et al, KogyoKayaku **31**(3), 131–7 (1970) & CA **78**, 6073(1973) (Combustion preventive chemicals for coal mine expls. I. Organic compds. Out of 60 compds tested only ammonium, sodiym or potassium oxalate, sodium or potassium formate, and calcium tartrate were effective)
95) Y. Matsuguma et al, KogyoKayaku **33**(4), 214–22 (1972) & CA **78**, 6074 (1973) (Combustion preventive chemicals for coal mine expls. II. Inorganic compds. Effective compds were aluminum nitrate nonahydrate, aluminum sulfate octadecahydrate and magnesium sulfate heptahydrate)
96) E. Samuelsen, GerP Disc! 2211527 (1972) & CA **78**, 6091 (1973) [Water-containing expl mixtures. An example is 87 parts of: a suspension of guar gum 0.1, tapioca starch 0.4, EGc 1.5 parts with a soln containing AN 40, calcium nitrate (calcium nitrate 79, AN 6, w 15%) 45, sodium nitrate 4, urea 5, water 4 parts at 65°; and 13 parts of: Polyhall 295 0.4 in EGc 75.2 parts at 80°, cooled, mixed with Al flakes 18 and powdered asphalt 9.4 parts; add 50% aq sodium dichromate 0.3 part when mixing the 87 and 13 parts to gel the mass]
97) P.E. Adams & P.W. Fearnow, USP 3678140 (1972) & CA **78**, 6093 (1973) (Foaming aqueous,

protein-containing, blasting agents. An example is to slurry AN, sodium nitrate, water, methylamine nitrate, sugar, coal, sulfur and egg albumin with guar gum and EGc followed by extrusion thru orifices at 40–160 psi into a suction chamber to give sp gr of 1.05–1.08)

98) W.F. Clark, USP 3695950 (1972) & CA **78**, 6094 (1973) (Amino-aldehyde resin blasting compn containing gaseous voids. An example is thiourea 10, sodium nitrate 10, AN 30, powdered Al 2½, 37% aq formaldehyde 10 parts, adjusted to sp gr 1.17 with glass microspheres)

99) W.F. Clark, USP 3695948 (1972) & CA **78**, 6095 (1973) (Cast expl compn containing thiourea. An example is water 4.8, thiourea 19.1, AN 57.0, sodium nitrate 19.1 %, adjusted to sp gr 0.90 with glass microspheres)

100) D.W. Edwards, USP 3695947 (1972) & CA **78**, 6096 (1973) (Aq expl comprising a higher amine, a gelling agent, and an inorganic oxidizer salt. An example is 60% aq AN 200, modified guar gum 2.8, ethoxylated octadecylamine 2 gm)

101) H.E. Mager, GerP Discl 2211635 (1972) & CA **78**, 18453 (1973) (Crystalline urea-containing expl mixture. Addition of AN 240, phenyldimethylamine 10, nitrophenol 10, and urea 500g to one liter of 50% nitric acid gives a non-hygroscopic expl mixture containing urea nitrate, polynitrophenol, polynitrophenyl dimethylamine, and AN and having deton vel of 3600–4200m/sec)

102) M. Vercauteren, USP 3684596 (1972) & CA **78**, 18454 (1973) (Pulverulent and water-bearing expl mixtures. Prepd from water 0–2, liq hydrocarbon 3–6, and AN-KNO_3 50–97%; the latter was prepd in the presence of fatty alkyl ammonium chloride and contributes low temperature detonability)

103) J.O. Goliger & R. Thiard, FrP 2102815 (1972) & CA **78**, 18456 (1973) (Expl compns comprising heterogeneous granules. Composed of 15–60% 1–5mm TNT, Pentolite, or Cyclotol and 40–85% 1–5mm w-sol oxidizer such as AN or sodium nitrate)

104) M. Kluensch et al, GerP Discl 2126920 (1972) & CA **78**, 45854h (1973) [Perchlorate-containing safety expls. An example is Al 25, AN 44.9, guar flour 0.1 and (methylamine-perchlorate 15, ethanolamine nitrate 43.4, methylamine nitrate 14.4, urea 14.4, AN 12.8%) 30%] 105) F.E. Slawinski, USP 3671342 (1972) & CA **78**, 60376x (1973) (Dynamite compns containing expanded thermoplastic beads. An example is beads 1, 4:1 Nitroglycol:Nitroglycerine 37.8, DNT 2.1, Nitrocotton 0.5, NC 0.3, AN 38.1, sodium nitrate 19.1, wood pulp 3.5, S 1.6 and chalk 1 part; such a mixture will explode under 500 feet of water)

106) M. Hamazaki & N. Nakajima, JapP 71 27397 (1971) & CA **78**, 74350 (1973) (Expl compns for blasting concretes. Safety expls consist of Al or Al-Mg alloy 20–40, KNO_3 20–50 and BaO 10–20 wt %)

107) E.A. Tomic, USP 3711345 (1973) & CA **78**, 99994 (1973) (Chemical foaming of water-bearing expls. Addn of under 0.1% of a borohydride to a typical gelled aq blasting agent containing a liquid carbonaceous fuel results in formation of stable 20–100 micron bubbles which sensitize the expl)

108) J.D. Chrisp, USP 3706607 (1972) & CA **78**, 113581 (1973) (Chemical foaming of water-bearing expls. Sensitizing 20–60 micron bubbles are created in a typical gelled aq blasting agent by in situ oxidation of hydrazine)

109) E.A. Tomic, USP 3713919 (1973) & CA **78**, 126468 (1973) (Chemical foaming of water-bearing expls with N,N′-Dinitrosopentamethylenetetramine. A typical gelled or emulsified aq blasting agent is sensitized by addition of the Dinitrosotetramine blowing agent to form 10–100 micron bubbles)

110) C.G. Wade, USP 3715247 (1973) & CA **78**, 126471 (1973) [Water-in-oil emulsion expl containing entrapped gas. An example is AN 31.7, $NaNO_3$ 10, Ethylenediamine Dinitrate 10, NH_4ClO_4 10, magnesium nitrate hexahydrate 12.3 and water 20 (heated until all dissolved), blended with a mixture of Indra 2119 wax 2 and sorbitan monooleate 10 parts, followed by addition of Corcel hollow glass agglomerates to make 100 parts]

EXPLOSIVES, PHYSICAL TESTS AND ANALYTICAL PROCEDURES ARRANGED IN CHRONOLOGICAL ORDER

In addition to the books, pamphlets, papers and reports listed in Vol **5**, pp D1649 & D1650 as *references*: 1, 2, 3, 4, 5, 6, 7, 8, 9, 9a, 10, 11, 12, 13, 13a, 13b, 13c, 13d, 14, 15, 16, 17, 18, 19 & 20 and under Foreign Methods of Examination, the refs 1, 2, 3, 4, 5 & 6, the following books, etc may be included as additional references:

A) A. Marshall, "Explosives", Churchill, London, Vol **2**(1917), pp 411 to 439: "Physical Characteristics and Tests"; 440 to 486: "The Pressure and Heat of Explosion"; 487–502: "Power and Violence of Explosion"; 503 to 532: "Ignition and Detonation"; 644 to 672: "Stability Tests"; 675 to 750: "Materials and Their Analysis"

B) E. de W.S. Colver, "High Explosives", Van Nostrand, NY(1918), pp 63–81 (Testing and Analyzing Raw Materials) and pp 228–40 (Testing and Analyzing Trinitrotoluene)

C) E. deB. Barnett, "Explosives", Van Nostrand, NY(1919), pp 169–206: "Explosive Properties Tests"; 207–226: "Sensitiveness and Stability Tests"

D) A. Stettbacher, "Schiess- und Sprengstoffe", Verlag A. Barth, Leipzig(1933), pp 361 to 375: "Die praktische Prüfungsverfahren für Sprengstoffe"

E) M. Giua, "Lezioni di Esplosivi, Parte Analitica", StabTipografico, L. Rattero, Torino (1933)

F) Coll, "Analisis y Pruebas de Pólvoras Sin Humo", Republica Argentina. Printed in Sweden in 1934. Part 1 – Tests, Chemical and Physical; Part 2 – Ballistic Tests. Comprehensive description of methods similar to those practiced by the Aktiebolaget Bofors Nobelkrut, Sweden (Complimentary copy supplied by Capitán Francisco O. Mauro of Fábrica Naval de Explosivos, Azul, Rep Argentina, to Senior author during his visit of Azul in 1955)

G) L. Wöhler & C. Wenzelberg, MAF **14**, 466–78(1935) (Essais Nouveaux sur La Sensibilité au Choc des Explosifs)

H) J. Reilly, "Explosives, Matches and Fireworks", Van Nostrand, NY (1938), pp 50 to 58: "Quantitative Analysis"; 65 to 70: "Physical and Explosive Tests"; 70 to 93: "Stability Tests"

I) J. von Meerscheidt-Hüllesem, SS **34**, 167–70(1939) & CA **33**, 7569(1939) (Determination of the chemical stability of explosives including smokeless propellants with the aid of Winkler gas buret)

J) "Thorpe's Dictionary of Applied Chemistry", Longmans-Green, London, Vol **4**(1940), pp 546 to 552 (Testing of Explosives)

K) E. Piantanida, "Chimica degli Esplosivi e dei Gas di Guerra", TipRAccademiaNavale, Livorno (1940), pp 302–29 (Various stability tests)

L) F.H. Goldman, JIndHygToxicol **24**, 121–22(1942) (Determination of content of expls, such as NG, DNT, TNT, Tetryl, PETN, etc in air by *impinger method* using suitable solvents)

M) G.D. Clift & B.T. Fedoroff, "A Manual for Explosives Laboratories", Lefax, Inc, Philadelphia, Vol **1**(1942): Chap I, Part 1 (Analysis of commercial mixed acids); Chap I, Part 2 (Analysis of oleums including those contg nitric acid); Chap I, Part 3 (Analysis of spent acids); Chap II, Part 1 (Laboratory practice); Chap II, Part 2 (Precise method of weighing); Chap II, Part 3 (A modified method for the detn of nitrogen in nitroaromatic compds); Chap IV (Analysis of toluene used in manuf of TNT); Chap V (Trinitrotoluene tests); Chap VII (Detn of impurities in crude TNT); Chap VIII [Properties and analysis of MNT's, DNT's, Di-Oil (Commercial DNT), TNT's and commercial TNT]; Chap IX, Pt 1 (Analysis of commercial nitric acid); Chap IX, Pt 2 (Analysis

of commercial sulfuric acid and oleum); Chap X [Specification requirements and and tests for DNT used in FNH (Flashless Non Hygroscopic) propellant]; Chap XI, Pt 1 (Nitroglycerine and NG explosives); Chap XI, Pt 2 (Tests for NG); Chap XI, Pt 3 (Tests for NG expls); Chap XII, Pt 1 (Nitrocellulose and smokeless propellants, including the tests for raw materials); Chap XII, Pt 2 (NC tests and smokeless propellants tests); Chap XII, Pt 3 (Additional tests to be applied to any propellant)l Chap XIII (Amatol) ; Chap XIV, Pt 1 (Picric Acid); Chap XIV, Pt 2 (Ammonium Picrate); Chap XV (Nitrostarch Explosives); Chap XVI, Pt 1 & Pt 2 (Tetryl); Chap XVII (Black Powder); Chap XVIII (Primers and detonators)

N) Ditto, Vol **3**. *Supplement Number One to Volume 1* (1943). Chap I (Analysis of acids including illustrations of Berl pipette and Weighing pipette used at Keystone Ordn Works, Meadville, Pennsylvania); Chap II (Various laboratory procedures); Chap IV (Analysis of toluene used for manuf TNT); Chap V (Analysis of TNT); Chap VI [Analysis of *Sellite* (Na sulfite soln) used for purification of crude TNT]; Chap VI [Analysis of "soda ash" (crude Na carbonate)]; Chap VI [Analysis of brimstone (crude sulfur) used for manuf sellite]; Chap VII [Analysis of Tri-Oil (crude TNT), yellow waters, and red waters formed during manuf of TNT]; Chap VII (Analytical procedures used at TNT waste disposal plant, described by Dr H.D. Minnig); Chap VIII (Detn of small amts of sulfuric acid in nitric acid and detn of small amts of nitric acid in sulfuric acid)

O) Ditto, Vol **3**, *Supplement Number Two to Volume 1* (1944). Chap I [Ammonia Oxidation Plant; Rapid method for estimation of ammonia in gases entering the converter; Laboratory control at Ammonia Oxidation Plant; Absorption tower efficiency; Ammonia oxidation conversion efficiency. Determination of small amounts of nitrogen oxides (Except N_2O & N_2O_3) in stack gases or in atmosphere]; Chap I (Pickling of platinum multilayer gauze); Chap I (Analyses of acids manufactured and used at TNT Plant); Chap I (Determination of efficiency of precipitators in Sulfuric Acid Concentration Plant); Chap II (Calibration of laboratory glassware); Chap II (Calibration of balances and weights); Chap IV [Determination of unsaturated compounds (olefins) in toluene]; Chap V (Determination of sodium in TNT); Chap VI (Analytical procedures conducted at "Sellite" Plant); Chap VII (Analytical procedures conducted at Waste Disposal Plant); Chap VII (Tetranitromethane; determination in crude TNT and in other organic compounds); Chap XIV (Analysis of fuel oils used at Explosives Plants); (Analytical procedures conducted at Power House, such as analysis of coal and water); Chap XX (Toxicity of explosives and tests)

P) A.L. Olsen & J.W. Green, "Laboratory Manual of Explosive Chemistry", J. Wiley, NY (1943), pp 1 to 8 (Safety in laboratory organization); 9 to 20 (Propellants raw materials; testing and inspection); 20 to 24 (DuPont nitrometer); 26 to 32 (Testing Nitrocellulose); 32 to 50 (Smokeless propellants and their testing); 51 to 58 (TNT and testing); 58 to 60 (Amatol and testing); 60 to 66 (Picric Acid and testing); 66 to 70 (Ammonium Picrate and testing); 70 to 74 (Nitrostarch and testing); 74 to 81 (Tetryl and testing); 82 to 86 (Primers, Igniters & Iniators and testing); 87 to 91 (Black Powder and testing) ; 91 to 95 (Mercury Fulminate and testing); 95 to 97 (Lead Azide and testing); 98 to 101 (Sampling)

Q) Kast-Metz (1944). See Ref 3 on p D1650-R of Vol 5

R) Vivas, Feigenspan & Ladreda **4** (1946), Chapters II to XIV. See Ref 5 on p D1650-R in Vol 5 of Encycl

S) A.M. Soldate & R.M. Noyes, AnalChem **19**, 442–4 (1947) & CA **41**, 6105 (1947) (X-ray Identification of explosives). Tables of X-ray interplanar spacings and intensities are given for the common morphological

forms of the following expls and non-expl additives of expls: PETN, 2,4-DNT, TNT, PA, Gu Picrate, EDNA, NGu; 2,2,5,5-Tetranitratomethylcyclopentanone; Dinitratoethylnitramine; Hexahydro-1,3,5-trinitro-s-triazine; Octahydro-1,3,5,7-tetranitro-s-tetrazine; 1-Acetyloctahydro-3,5,7-trinitro-s-tetrazine; α,β-Diethylcarbanilide, DPA, N-nitroso-DPA, 2-nitro-DPA; 2,4-dinitro-DPA; & 4,4′-Dinitro-DPA

T) L. Médard & M. Thomas, MP **31**, 173–196 (1949) (Colorimetric determinations of 28 nitrocompounds and organic nitrates) [Included were: 2,6-DNT; 4,6-Dinitro-o-cresol; 2,6-Dinitro-p-cresol; 2,4,6-Trinitroaniline (Picramide); sym-Trinitromesytylene (1,3,5-Trimethyl-2,4,6-trinitrobenzene); 2,4,6-Trinitroresorcinol (Styphnic Acid); 2-Amino-4,6-dinitrophenol; 2,4,6-Trinitrobenzoic Acid; 2,4,6,2′,4′,6′-Hexanitrodiphenylamine (Hexyl); 2,4,6-Trinitrophenylethylnitramine; Bis-(2,4,6-Trinitrophenyl)-ethylenedinitramine (Bitetryl) ; Cyclotrimethylenetrinitrosamine; Ammonium Picrate; Nitroguanidine; Nitrourea; Urea Nitrate; Ethylenediamine Dinitrate and Nitro-iso-butanetriol Trinitrate]

U) C. Caprio, "Corso di Esplosivi", Scuola Salesiana del Libro, Rome, Vol **2**(1949), 184–236 (Various stability tests); 238–39 (Chemical tests); 239–43 (Physical tests); 246–50 (Ballistic tests)

V) L. Médard, MP **33**, 323–55 (1951) & CA **47**, 10227 (1953) (Explosives, Testing). Brief descriptions of standard tests are given, along with typical values for important expls

W) C. Belgrano, "Gli Esplosivi", U. Hoepli, Milano (1952), pp 15 to 53 (Physical tests for mining explosives. Included are: density, stability, Trauzl test, detonation velocity, Hess test, crusher test, sympathetic detonation test, sensitiveness to initiation, sensitiveness to impact, steel plate test and explosion temperature determination); pp 73 to 75 (Analysis of NG); 76 to 84 (Analysis of acids in NG manuf); 93 (Analysis of NGc); 96 to 97 (Analysis of PETN); 105 to 108 (Analysis of Collodion Cotton); 114 to 115 (Analysis of propellants); 119 (Analysis of DNT); 129 (Analysis of TNT); 135 (Analysis of RDX, called T_4 in Ital is conducted as for PETN described on pp 96–97); 161–68 (Analysis of AN and its explosives); 168 to 171 (Galleries for testing permissible expls); 171 to 172 (Measurement of duration and length of flame produced on explosion)

W_1) Kirk & Othmer, Vol **8** (1952), pp 654–57 (Section on Testing Exolosives which includes: Impact Tests, Explosion Temperature Tests, Sensitivity to Initiation, Pendulum Friction Test, Rifle Bullet Test, Sand Test, Rate of Detonation Determination, Ballistic Pendulum Test, Trauzl Lead Block Test, Heat of Explosion, 65.5°C Surveillance Test, 75°C International Test, 100° Heat Test and Vacuum Stability Test. No tests are described in 2nd Edition of K&O

X) P. Aubertein, MP **37**, 139–52 (1955) (Particle size measurements of expls) (Granulométrie)

Y) H. Koenen & K.H. Ide, Explosivstoffe **3**, 89–93 (1955), 9 refs (Testing of explosives)

Z) P. Aubertein, MP **38**, 29–37 (1956) (Analysis of phlegmatized explosives)

AA) H. Koenen & K.H. Ide, Explosivestoffe **4**, 119–25 & 143–48 (1956), translated in 1959 by G.R. Loehr & Henry Voos, PicArsn, FREL Transln No 26 (Steel-Container Method for Testing Explosive Materials)

BB) Frank Pristera et al, PATR **2254** (1956) (Analysis of explosives by IR spectrum method)

CC) L.L. Vagnina, MikrochimActa **1956**, 221–25 & CA **50**, 8401 (1956) (Identification of expls in legal chemistry)

DD) M.A. Cook, "The Science of High Explosives", Reinhold, NY (1958). Tests described by Cook include: pp22–29 (Wave-Speed Photography); 29–31 (Pin Method); 29–32 (Microwave Method); 32–35 (Meas of Deton Press); 35–36 (High Speed Photography); 36–37 (Available Energy); 37–38 (Temp of Deton); 38–39 (Impact Sensitivity); 39–41 (Thermal Decomposition); 41 (Miniature Charge Techniques); 42 (Charge Preparation)

EE) H. Koenen et al, Explosivstoffe **6**, 178–89, 202–14 & 223–34(1958) (Testing of expls) (42 refs)

FF) P. Aubertein & H. Pascal, MP **40**, 113–25(1958) (Analyses of expls and their starting materials) (54 refs)

GG) L. Médard, "Physics of Explosives", Series of articles with numerous illustrations and refs in MAF **31**, 553–83(1957); **32**, 909–52(1958); **33**, 7–58(1959); **34**, 387–417, 585–622, 899–946(1960); and **35**, 263–332 & 509–62(1961)

HH) M. Roth & R.E. Wegman, AnalChem **30**, 2036(1958). A soln containing NGu is analyzed for the NGu content by addition to excess deaerated titanous chloride followed by back titration with standard ferric ammonium sulfate

II) M. Roth, M. Laccetti & M.R. Youngiver, PicArsnTechMemo **GL-8-59**(1959). "Abridged Spectrophotometric Method for the Determination of Available Stabilizer Content and Application to Prediction of Safe Life of Propellants"

JJ)M.A. Laccetti, S. Semel & M. Roth, AnalChem **31**, 1049–50(1959). The ferrous sulfate-sulfuric acid method is used to differentiate HMX and RDX from NGu, Tetryl, TEGN, PETN, NG & K nitrate. Following the procedure given (which requires the colorimetric measurement of the color produced on dissolving the sample in the reagent) reveals that HMX and RDX have Beer's Law curves which differ from one another and from the single curve which the other six compds follow. By testing different amounts of sample to generate a curve it is possible to identify the HMX or RDX and quantitatively measure it by comparison with a standard for each

KK) Michele Giua, "Trattato di Chimica Industriale", UTTET, Torino, **6**(1)(1959) [Includes on pp 433 to 463 – Physical Tests, which include: impact test, Trauzl test, sand test, sensitivity to initiation, decomposition temperature; detonation velocity by methods of LeBoulangé, Dautriche and Mettegang; sympathetic detonation or gap test; rifle bullet test; length and duration of flame test; gallery tests; specific weight determination; heat tests, such as of Abel, Angeli, Bergmann-Junk, Waltham Abbey (Silver vessel test); Grottanelli, Guttmann, Hess, Hoitsema, Mayrhofer, Obermüller, Sy, Taliani, Thomas, Vieille and Will. On pp 463 to 475 are Chemical Analyses of various explosive compds, explosive compns and propellants. On pp 475 to 488 are Microscopic Analyses which include 72 microscopic photos of crystals. On pp 488 to 494 is given a brief description of analyses of acids used in nitration, of raw materials and of nonexpl substances used as ingredients of various expl compns]

LL) F. Pristera et al, PATR **2254**(1956) (Analysis of Explosives by Infrared Spectroscopy). A compilation of 68 infrared spectrograms covering all common HE's and many possible expl ingredients, additives and related compds. In the analysis of single or multi-component HE's, infrared spectroscopy is, in most cases, very useful either alone or in connection with other methods. Infrared spectroscopy has the advantages of speed, specificity and very often accuracy & precision

MM) J. Sinabell, Explosivstoffe **7**, pp 7 & 141–44(1959) (Determination of CO_2 and N oxides in explosive vapors)

NN) Anon, "Analytical Methods for Powders and Explosives", AB Bofors, Nobelkrut, Sweden (1960) [pp 42–60 – Stability tests; 60–62 – Explosion temperature test; 63–65 – Explosive character tests, which includes Trauzl-, impact-, and detonation velocity tests; 67–155 – Examination of raw materials for propellants, explosives and pyrotechnics; 159–164 – Examination of nitrated products; 165 – Examination of nitration acids; 167–191 – Examination of finished products: propellants, secondary expls and primary expls; 192 – Examination of individual expls: solid TNT, liquid TNT, Hexogen (RDX), Hexotol (Cyclotol), Hexotonal (RDX/TNT/Al, Torpex), Penthrite (PETN), Bofors Plastic Explosive (BPE), Bonocord, Tetryl, Lead Azide, Lead Styphnate, Mercury Fulminate, Silver Azide and Tetracene]

OO) M. Roth of Picatinny Arsenal, "Current Accomplishments on Analytical Chemistry Project on Evaluation and Establishment of Methods of Analysis of Propellants, Explosives and Chemical Ingredients", Presented at Third Meeting of Operations Subcommittee to the Integration Committee on Propellants and Explosives, US NOTS, China Lake, Calif, 16 March 1960 [Following are some accomplishments: a) Modification of DuPont nitrometer by introducing a 2-part bulb. This bulb is recommended for use with PETN and Nitroguanidine (NGu) (abbr NQ by Roth): This modification is described by M. Roth et al in PATR **2579**(1958); b) Introduction of "titanous chloride-buffer method" for detn of NGu in propellants. This method is described by M. Roth & R.F. Wegman in AnalChem **30**, 2036(1958) (See Addnl Ref HH); c) Improved method for detn of stabilizer content. The method is described by M. Roth et al in PicArsnTechMemo **No GL-8-59** (See Addnl Ref II); d) Improved *colorimetric method* of analysis of binary mixtures of nitrate and nitramine or nitramine alone. This method described by M.A. Laccetti et al in AnalChem **31**, 1049–50(1959) gave good results in analysis of RDX-HMX mixtures (See Addnl Ref JJ); e) Development of a volumetric method for RDX as well as other expls, based on *reduction by chromous chloride.* A noteworthy feature of methods employing this reagent is that the reaction proceeds at room temp. The description of method was not given in open literature as of 1960; f) Among the instrumental methods used at PicArsn for quality control work, *absorption spectrophotometry* was found the most useful type of measurement. Some spectra have been published by F. Pristera et al in PATR **2254**(1956) (See Addnl Ref LL); g) Evaluation of *tetraphenylboron method* for the determination of potassium]

OOa) Ye.Yu. Orlova, "Khimiya i Tekhnologhiya Brizantnykh Vzryvchatykh Veshchestv" (Chemistry and Technology of High Explosives), GosIzdatOboronProm, Moscow (1960) (No tests for explosives or propellants are described)

PP) J.W. Frazer, "A New Explosive Compatibility Test", UCRL **6244**(1960). See Ref CD

PPa) A.H. Rosen & H.T. Simmons, NAVORD Rept **6629**(1959), US Naval Ordn Lab, White Oak, Md. This rept describes an improved constant temp block used to study the stability & compatibility of expls at elevated temps. A very small area of the heating block is exposed while introducing or removing a sample from any one of 12 sample positions; achieved by means of a rotating top. A single piece all glass unit consisting of a sample tube & manometer is described for making vacuum stability measurements at elevated temps

QQ) Z.G. Pozdnyakov, "Testing the Stability of Industrial Explosives" (in Russ), IzdatInstGornDela, Moscow (1961)

RR) E.J. Murphy, "Comparison of Methods for Detecting and Analyzing Fumes from Explosives", USBurMines **RI 5833** (1961). A comparative study of several analytical methods for determining carbon monoxide and nitrogen oxides in toxic gases produced by detonation of explosives

SS) I. Dunstan & J.V. Griffiths, "Determina-

Course in the Theory of Explosive Substances" (in Russ), RosVuzIzdat, Moscow (1963)

AB) F.J. Welcher Edit, "Standard Methods of Chemical Analysis", VanNostrand, NY, Vol 2, Pt 2 (1963), pp 454–504, Organic Analysis by Siggia

BC) F.J. Welcher, Edit, "Standard Methods of Chemical Analysis", VanNostrand, NY, Vol 2, Pt 3 (1963), Chapter 32, pp 1283 to 1410, "Explosives and Propellants" by Raymond H. Pierson of US Ordnance Test Station, China Lake, Calif. It includes:
Black Powder Tests (p 1285)
General Methods (pp 1286 to 1305)
Heat of Explosion Determination (1286–88)
Hygroscopicity of Propellants (1288–89)
Moisture by the Carbon Tetrachloride Distillation Procedure (1289–90)
Moisture by Karl Fischer Titration (1290–93)
Moisture and Volatiles by Oven Drying at 100° (1294)
Moisture and Volatiles by Vacuum-Oven Drying at 55° (1294)
Nonaqueous Titration (1294–95)
Stability by International Test at 75°C (1295)
Stability by Potassium Iodide-Starch Test. See pp 1333 under "Stability Test: Heat Test with Potassium Iodide-Starch Test"
Stability by Heat Test at 120° or 134.5°C With Standard Methyl Violet Paper (1295–96)
Stability by Surveillance Test at 65.5°C (1296)
Stability by the Taliani Test for Propellants (1296–97)
Stability by Vacuum Stability Test for Propellants or Ingredients (1298–99)
Strand Burning Rate of Solid Rocket Propellants (1299–1304)
Total Volatiles by the Solution Evacuation Procedure. See footnotes 29 & 30, p 1375
Titrimetric Calculations (1304–05)
Ingredients of Explosive and Propellant Compositions (1306 to 1344):
Aluminum (1306–07)
Silicon (as Si) (1308)
Zinc (1308)
Iron (1308–09)
Magnesium (1309)
Copper and Lead (1309–10)
Nickel (1310–11)
Manganese (1311)
Ammonium Nitrate (1311–13)
Ammonium Perchlorate (1313–16)
1-1-(or unsym)-Dimethylhydrazine (UDMH) (1316–17)
Hydrazine (1317–18)
Hydrogen Peroxide (1318–21)
Magnesium, Powdered (1321–23)
Magnesium Oxide (1323–25)
Nitric Acid, Fuming (White or Red) (1325–30)
Nitrocellulose (Cellulose Nitrate) (1330–35)
Nitroglycerin (Glycerin Nitrate) (1335–36)
Nitroguanidine (Picrite) (1336–37)
Propellants (1371–1407)
Polypropylene Glycol (1337–39)
2,4-Toluene Diisocyanate (1339–40)
Triacetin (Glyceryl Triacetate) (1340–41)
2,4,6-Trinitrotoluene (TNT) (1341–43)
Zinc Oxides (1343–44)
Quantitative Tests of Explosives (1345 to 1370)
Lead Azide (LA) (1345–46)
Mercury Fulminate (MF) (1346)
Diazodinitrophenol (DAzDNPh or DDNP) (1346)
Lead Styphnate (LSt) (1346)
Tetracene (1346–47)
Noninitiating Explosives, General Tests (1347)
Auxiliary Tests for Specific Materials: RDX, Composition A-3, Comp B, Comp C, Torpex, Tritonal, Amatol, Ammonal (1347 & 1349). Colorimetric tests for some expls are on p 1348
Black Powder Initiating Explosive as Used as an Igniter or Fuze (1349–50)
DYNAMITES (1350–64)
Blasting Caps and Electric Detonators (1365–67)
Primers (1367–70)
Nitrocellulose-Base Solid Propellants, Including Single-Base, Double-Base and Triple-Base Propellants (1371–1405)
Composite Propellants (1405–07)
Appendix A. Partial List of Specifications and Abbreviations Related to Explosives, Propellants, or Their Ingredients (1408–10)

CD) J.W. Frazer & K. Emst, "Chemical Reactivity Testing of Explosives", Explosivstoffe 12, 4–9 (1964) [The following tests are described: a) Determination of NO, N_2O & NO_2 in decomposition products of

tion of Carbon Content by Wet Combustion: Application to Explosives and Initiators", ERDE Tech Memorandum No **2/M/62**(1962), OTIA 18588

TT) E. Haeuseler, Explosivstoffe **10**, 97–104 (1962). Melting point apparati are described and compared in determining the mp's of various expls and non-expls. The Thiele Tube, which uses a liquid or molten salt bath surrounding a capillary tube containing the substance and attached to a thermometer, is represented by the Tottoli improvement; this incorporates stepless internal electrical heating and a stirrer. The Maquenne Block represents the electrically heated metal surface (2.6 x 25cm) type, on which one places the substance; to hold down sublimation and decomposition effects, the block is heated to the vicinity of the expected mp, and then samples of the substances are periodically placed on the block as the temp is further raised. The Kofler Block is a metal surface 3.8 x 37cm with a fixed temperature gradient of 50–260°; the substance is linearly spread on it in the expected mp region. Accuracy with the latter is considerably lower than other methods, only ± 0.4–1.0°. When small amounts of the substance are available, the Hilbck Micro Hot Stage is used. The substance is placed between cover glasses sandwiched between two heated metal discs, both having holes in the center. A low power microscope is used to observe the mp thru the upper hole, illumination entering from the lower. Except for the Kofler method a collection of narrow range thermometers is required for careful work

UU) A.R. Lusardi, "A Manual of Laboratory Procedures for Analysis and Testing of Explosives and Pyrotechnics", Explosives and Propellants Laboratory, FREL, PicArsn, Dover, NJ, 1962
Section 100 (Methods 101 to 118). Specification Tests of Single Component Explosives: Ammonium Picrate (Explosive D), Diazodinitrophenol (DAzDNPh, Dinol, Diazol), Dinitroethylbenzene (DNEB or DNEtBz), Dinitromethylaniline (DNMA or DNMeAn), DNMA in Sulfuric Acid, Dinitrotoluene (DNT), Halite (EDNA), HMX (Cyclotetramethylenetetranitramine), Lead Azide (LA), Lead Styphnate, Basic (LSt,B), Lead Styphnate, Normal (LSt,N), Mercury Fulminate (MF), Nitroguanidine (NGu, Picrite), Pentaerythritol Tetranitrate (PETN), RDX (Cyclonite, Cyclotrimethylenetrinitramine, Hexogen), Tetranitrocarbazole (TeNCbz, TeNC), Tetryl (Trinitrophenylmethylnitramine) and TNT (Trinitrotoluene Trotyl)
Section 200 (Methods 201 to 212). Specification of Multiple Component Primer Compositions: Fuze Powder; Primer Mix, US Standard; Primer Mix, New No 4; Primer Mix, MK V; Primer Mix, No 70; Primer Mix, PA100 Primer Mix, NOL No 130; Primer Mix, M3 Ignition Cartridge; Primer Mix, M29 Percussion Primer; Priming Composition, Lead Starter Type; RDX Booster Pellets; and Tetryl Booster Pellets

VV) Anon, "Military Standard. Explosive: Sampling, Inspection and Testing", **MIL-STD-650**, US GovtPtgOffice, Washington, DC (1962). Included are 81 methods which are subdivided into:
Group 100: General Test Methods
Group 200: Physical Test Methods
Group 300: Sample Preparation
Group 400: Chemical Test Methods
Group 500: Stability Sensitivity and Surveillance Tests
Group 600: Standard Solutions
Group 700: Standard Indicators

WW) K. Trautzl, Explosivstoffe **11**, 71–3 (1963) (Measuring brisance by compression)

XX) A.F. Williams & D. Kenyon, Explosivstoffe **11**, 249 (1963) (Application of cathode-ray polarography to analysis of mining explosives)

YY) M. Leclercq, MP **45**, 208–14 (1963) (Identification of explosives by their residues of combustion. Infrared spectrography method)

ZZ) A. Gol'binder, "Laboratory Works for a

expls & proplnts; b) Determination of acidity; c) Pressure and volume change measuremants; and d) Weight loss determinations]

CD_1) T. Urbański, Vol **2**(1965), pp 22–31 gives methods for determining the Stability of Nitric Esters, which include: Abel Heat Test, Litmus Test (Vieille Test), Heat Test at 134.5°C, Silvered Vessel Test, Loss of Weight Test, Will Test, Bergmann & Junk Test and Manometric Test

DE) A.A. Menegus, Ordn **49**, 86–8(1964) (Explosives, Testing by TV). Closed circuit TV is used at Picatinny Arsenal to observe laboratory prepn of new expls, mechanical shaping of solid expls, field testing of ordnance items, recovery and disposal of misfires, etc. By remote control the camera lenses may be directionally oriented, especially in conjunction with operation of remote control "slave" devices and vehicles

EF) C. Giorgio, "Tecnica degli Esplosivi", Del Bianco, Udine (1964), Chap IV, *Physical Tests* (pp 41 to 56), which include: Detonation velocity (42–8); Pressure of explosion (49–53); Volume of gases of explosion (53–4); Heat of explosion (54–6). *Chemical Analyses* (56–58), which include: determinations of mp, impurities, acidity, alkalinity, carbon by combustion, and separation of ingredients by various solvents. *Stability Tests* (56–66); Hygroscopicity (66); Freezing (66); Exudation (67); Explosion temperature (67–8); Sensitivity to flame and to high temperature in general (68–9); Sensitivity to impact (70–2); Sensitivity to initiation (72–3); Efficiency of initiators (73–4); Sensitivity to rifle bullet (74); Power of explosives by Lead Block Test (75–81); Brisance by Hess' method (81–2); Brisance by Hess' modification (82); Brisance by Kast's method (82–3); Swedish test for use in mining (86); Ballistic pendulum and Ballistic mortar (86–93); Ballistic pendulum of Schmidt (93); Ballistic pendulum for measuring impulse and shattering (93–4); Steel plate breakage test (94–5); Gap test (95–6); Gallery tests for permissible explosives (esplosivi grisoutosi) (96–8); Study of flame produced on expln (98–100); Propagation of detonation wave (100–101)

FG) W. Selig, "Some Analytical Methods for Explosives and Explosive Simulants", **UCRL-7873**, pp 1–65 (1964) & CA **64**, 9497–99 (1966). Describes the estimation of RDX & HMX by nonaqueous titrimetry; analysis of HMX, Poly (2,2-dinitropropyl-acrylate, silica, and ethyl & methyl 4,4-dinitropentanoate in expls; determination of PETN & silica in LX-02-1; spectrophotometric detn of acetyl tributyl citrate in extrudable expl LX-02-1; analysis of a mixt of HMX, Viton & oxamide; analytical procedure for RDX in RX-05; analysis of tungsten & HMX in RX-12; analysis of a mock expl 90010; analysis of a mock material for LX-0-4-1; and determination of cyanuric acid, melamine, and Viton in mixt LM-04-0

GH) A.J. Clear, "Standard Laboratory Procedures for Determining Sensitivity, Brisance and Stability of Explosives", PATR **3278** (1965), FREL, Picatinny Arsenal, Dover, NJ. The following tests are described: Impact Test with PicArsn Apparatus (pp 2 to 4 with Figs 1, 2, 3 & 4 on pp 32 to 35); Impact Test with USBurMines App (4 to 7 with Figs 5, 6 & 7 on pp 36 to 38); Modified Impact Tests for Liquid Explosive Made with BurMinesApp and with PicArsnApp (7); Explosion Temperature Test (7 to 8 with Fig 8 on p 39); Decomposition Temperature Test (8); Sensitivity to Initiation as Determined by Sand Test (9–11 with Figs 9, 10, 11 & 12 on pp 40–3); Modified Sand Test for Liquid Explosives (12–14); Electrostatic Sensitivity Test (14–15 and Figs 13 & 14 on pp 44 & 45); Brisance by Sand Tests (16–17); Initiation Efficiency by Sand Test (17); Stability Tests, which include: 75°C International Test (18); 82.2°C KI Test (19); 100° Heat Test (19); 90°, 100° & 120°C Vacuum Stability Tests (19–22 and Figs 15, 16 & 17 on pp 46–8); 65.5°C Surveillance Test (22–3 and Fig 18 on p 49); 80°C Surveillance Test (23); 120° and

134.5°C Heat Tests (23–5); Taliani Test (25–7 and Fig 19 on p 50); 65.5°C KI Test (27–9 and Fig 20 on p 51); and Reactivity Tests (29–31)

HI) S. Fordham, "High Explosives and Propellants", Pergamon Press, NY (1966), *Chapter 6*, "Assessment of Explosives", which includes: Power or Strength (pp 65–8); Velocity of Detonation (69–72); Sensitiveness to Friction and to Impact (72–6); Stability on Storage (76–8); Fume Test (78–9); and Miscellaneous Tests, such as for Brisance and Speed of Gelatinization of NC (79–80). *Chapter 7:* Gallery Testing of Permitted Explosives (85–9)

IJ) Anon, "Fuze and Fuze Components, Environmental and Performance Tests for", **MIL-STD-331** (1966) and Change Notices: **1**(1967), **2**(1967), **3**(1969), **4**(1971), **5**(1971) and **6**(1972)

JK) S.A.H. Amas & H.J. Yallop, Analyst **91**, 366–67 (1966). See Ref 13a in Vol **5** of Encycl, p D1649-R

KL) Anon, "Military Explosives", **TM 9-1300-214/TO 11A-1-34** (1967). *Chapter 5.* Properties and Tests of High Explosives: Sensitivity to Frictional Impact (pp 5-1 to 5-3); Sensitivity to Friction (5-3 to 5-6); Sensitivity to Frictional Impact (5-6); Sensitivity to Heat and Spark, which includes Explosion Temperature Test (5-6 to 5-9); Sensitivity to Initiation (5-9); Stability Tests, which include 75° International Test, 100° Heat Test, Vacuum Stability Test and Potassium Iodide–Starch Test (5-9 to 5-15); Brisance Tests which include Sand Test, Plate Dent Test and Fragmentation Test (5-15 to 5-18 and Fig 5-13 on p 5-19); Initiating Value (5-18 & 5-20); Sympathetic Detonation (5-20 to 5-21); Power which includes Heat of Explosion Test, Ballistic Pendulum Test and Trauzl Lead Block Test (5-21 to 5-24); Blast Effect (5-24 to 5-27); Cratering Effect (5-28 & 5-29); and Munroe-Neumann Effect (5-29 to 5-35)

LM) Anon, "Military Standard, Propellants, Solid: Sampling, Examination and Testing", **MIL-STD-286B** (1967) and Notice **1** (1969) & Notice **2** (1971)

MN) D.P. Lindstone, "The Assessment of Explosive Performance by Means of a Cartridge Case Deformation Test", Explosivstoffe **17**(9), 193–201 (1969). Abstracted in Expls&Pyrots **3**(8), 1970 [Two-gram charges are fired in brass small-arms cartridge cases (cal .303) and results compared with a cartridge in which a 2-g "standard" was fired]

NO) Encyclopedia of Explosives, PATR **2700**, Vol **4**(1969), pp D1060 to D1078, "Analytical Procedures for Explosive Compositions Used in Detonators, Primers, Igniters and Fuzes"; D1078 to D1107 – "Physical Tests for Determining Explosive and Other Properties of Detonators, Primers, Igniters and Fuzes"

OP) J. McCormack et al, "A New Procedure for the Estimation of the Impact Sensitiveness of Explosives", Explosivstoffe **17**(10), 225–28 (1969). Abstracted in Expls&Pyrots **3**(8), 1970 (Measurement of the Figure of Insensitiveness for relatively insensitive expls by the drop weight method is improved by detecting gas evolved in "no-fires" with starch-iodide impregnated filter paper. Quantities of gas less than 1 cc are readily detected)

PQ) M.F. Zimmer & L.D. Lipton, "Dynamic Pressure Measurements on Small Amounts of Detonating Lead Azide", Explosivstoffe **18**, 12–15 (1970). Abstracted in Expls&Pyrots **3**(8), 1970. (To evaluate the safety of handling small (few mg to 1 g) amts of LA in daily pilot plant operations, dynamic pressure measurements were taken with/or without protective cloth. The distance from the sample to the gauge was changed from 1 to 30cm)

QR) M. Cruchaud, "On the Sensitivity of Explosives to Mechanical Effects", Explosivstoffe **18**(1), 16–19 (1970) (in French). Abstracted in Expls&Pyrot **3**(8), 1970 (Various theories attempting to explain the initiation of expls by a falling weight are briefly presented along with refs to reported explns

under other circumstances which suggest a multiplicity of causative effects. The author then proposes that the underlying basis for any initiation is the attainment of a local electrical polarization in the expl which meets the "critical" value for that expl in the particular conditions at hand)

RS) R. Jenkins & H.J. Jallop, "The Identification of Explosives in Trace Quantities on Objects Near an Explosion", Explosivstoffe **18**, 139–41 (1970). Abstracted in Expls&Pyrots **4**(3), 1971 (Samples are taken by rubbing the object with cotton wool soaked in a solvent appropriate for the suspected expl, then extracting the cotton with more or a different solvent. The concentrated extract is then chromatographed on a thin layer of silica gel along with samples of known expls. The developed chromatograph is dried and then sprayed with any of four reagents which give distinctive colors for MNT, DNT, TNT, NG, AN, NGu, NC, RDX, HMX, PETN, or Tetryl. The color, in combination with the position of the colored spot on the chromatograph, will define the expl)

ST) F. Pristera of Picatinny Arsenal, "Explosives" in "Encyclopedia of Industrial Chemical Analysis", Vol **12**, J. Wiley & Sons, NY (1971). Reviewed in Expls&Pyrots **6**(1), 1973. Was listed as Ref 20 in Vol **5** of Encycl, p D1650-L. Here is a more detailed listing of contents by pages:
405–408, Handling of Explosives
406 & 407, Table 1. Properties of Primary Explosives
408–10, Mercuric Fulminate (MF) and Analyses
410–13, Lead Azide (LA) and Analyses
413–16, Lead Styphnate (LSt) and Analyses
416–17, Diazodinitrophenol (DAzDNPh) and Analyses
418–Table 7, Interplanar Spacings of Primer Ingredients in Approximate Order of Intensity, Å
419–21, Characteristics and Reactions of Individual Primer Ingredients
421–24, Analysis of Unknown Primer Compositions
424–31, Determination of Components in Known Primer Mixtures
424, Table 8, Composition of Ten Typical Primer Mixtures
431–51, High Explosives
432–33, Table 9. Physical Properties of Commercially Important High Explosives
446–50, Table 20. Common High Explosive Mixtures
451–52, Salts (Ammonium Picrate, Nitrate and Perchlorate)
452–60, Method of Analysis by Infrared Spectroscopy
460–62, Analysis of Unknown High-Explosive Mixtures
461, Table 22. Ingredients in Order of Increasing Melting Points
462, Table 23. Solubility of High Explosive Ingredients in Four Selected Solvents
463–65, Nitrogen Content of Nitrate Esters by Gas Evolution Using DuPont Nitrometer
465–66, Nitrogen Content of Nitrate Esters by Ferrous Reduction
466–67, Nitrogen Content of Nitro Aromatics by Titanous Reduction
467–70, Other Methods for Quality Control (Ash Content, Moisture, Bulk or Apparent Density, Specific Gravity by Mercury Displacement, and Solidification Point)
470–71, Sixty-seven References

TU) S. Semel, "Detection of Concealed Explosives", SMUPA-FR-E-C, PicArsn, Dover, NJ (1973). Reviewed in Expls&Pyrots **6**(4), 1973 (An experimental study has been completed on the feasibility of fast neutron activation for detecting concealed explosives in baggage. The same approach was first investigated by the North American Rockwell Corp, Los Angeles for the Federal Aviation Administration. The latter work was directed to the measurement of gamma radiation produced by activation of nitrogen in explosives. However, its feasibility for explosives detection is severely limited by the large number of false alarms due to the activation of common components in luggage which results in the same radiation as that from nitrogen. The method has now been extended by measuring the gamma rays from the neutron activation of both nitrogen and oxygen using two distinct detector channels. In this manner,

the probability of detecting the presence of an explosive is greatly increased with a corresponding decrease in the false alarm rate

UV) A.G. Gorst, "Porokha i Vzryvchatyiye Veshchestva" (Propellants and Explosive Substances), 3rd ed, IzdatMashinostroyeniye, Moscow (1972). *Chapter II.* Stability and Methods of Its Determination, which include: Abel Test (pp 32–3); Vieille's Test (33); Loss of Weight Test (33); Manometric and Electrometric Tests (34). *Chapter III.* Initiation of Explosives by the following methods: Heat or Flame (38–9 with Table 5 on p 39 giving Ignition Temperatures of various expls); Initiation by Mechanical Action, such as Impact Test (40–3); Friction Sensitivity by Method of Bowden-Kozlov (43–4); Initiation by Blasting Caps and Detonators (44); Detonation by Influence by Gap Test (44–5). *Chapter IV:* Heat of Explosion (49–51); Temperature of Explosion (51–3); Volume of Gases Produced on Explosion (58–9); Pressure of Gases Produced on Combustion and Explosion (59–61). *Chapter VI.* Determination of Detonation Velocity by Dautriche Method (80–1); Determination of Capacity for Work (Fugasnoye Deystviye or Fugasnost' in Russ) (Fugacity) . He uses for these tests Ballistic Pendulum, Trauzl Block, Cratering Action and Explosion of Artillery Projectiles (88–91); Brisance by Compression of Lead Cylinder (92–3); Action of Shaped Charge Explosions (Kumulyativnoye Deystviye Vzryva, in Russ) (93–5)

VW) Encyclopedia of Explosives, PATR **2700**, Vol **5**(1972), pp D1616-R to D1650-R – "Dynamites and Their Substitutes. Examination Including Analytical Procedures". Comprehensive description of methods used at US Bureau of Mines, DuPont Co, Hercules Co and Swedish AB Bofors, Nobelkrut. Also methods described by R. Pierson in Std-MethodsChemAnalysis, Vol **2**, Pt 2(1963)

WX) B.J. Alley & H.W.H. Dykes, JChromatogr **71**(1), 23–37(1972) & CA **77**, 128624(1972). Gas-liquid chromatographic determination of nitrate esters, stabilizers, and plasticizers in Nitrocellulose-base propellants

XY) J.W. Grindlay, AnalChem **44**(9), 1676–8 (1972) & CA **77**, 154615(1972). Determination of Hexahydro-1,3,5-trinitro-s-triazine (RDX) in Octahydro-1,3,5,7-tetranitro-s-tetrazocine (HMX) by infrared spectrophotometry

YZ) H. Bartels, Explosivstoffe **20**(7–8), 143–50(1972) & CA **78**, 126457(1973). Modified Gap test for the determination of the shock wave sensitivity of igniting and initiating materials

ZA) G. Norwitz & D.E. Chasan, Talanta **20**(1), 73–9(1973) & CA **78**, 99941(1973). Infrared macro- and semimicrodetermination of nitrogen in raw Nitrocellulose and Nitrocellulose contained in propellants

There are also descriptions of physical tests and analytical procedures under explosives of military or commercial interest and of raw materials. If any US Military Specifications are issued, their requirements and tests are described

Following is a selected list of US Military Specifications, arranged in alphabetical order:

TABLE OF US SPECIFICATIONS

Material	Abbreviation or Formula	Specification	Date
Acetic Acid	AcOH	JAN-A-465	April 1947
Acetic Anhydride	Ac_2O	JAN-A-459	March 1947
Acetin or Triacetin	TAc	JAN-T-301A	Dec 1961
Acetone, Technical	Acet	O-A-51F	July 1969
Acid, Acetic	AcOH	JAN-A-465	April 1947
Acid, Mixed	MA	MIL-A-50210	Dec 1968
Acid, Nitric	NA	JAN-A-183(2)	Oct 1951
Acid, Picric	PA	JAN-A-187	Feb 1945
Acid, Sulfuric	SA	JAN-A-179	Jan 1945
Adhesive & Sealing Compounds, Cellulose Nitrate Base, Solvent Type (For Ordnance Use)		MIL-A-82484	June 1967
Alpha-Nitronaphthalene (For Use in Explosives)	α-MNN	MIL-A-23894	Oct 1963
Aluminum Powder, Spherical	Al	MIL-A-81335	Nov 1965
Aluminum Powder, Spherical	Al	MIL-A-23950A	Sept 1966
Aluminum, Powdered, Flaked, Grained, and Atomized	Al	MIL-A-512A(2)	May 1967
Ammonia, Technical	Amm	O-A-445A	Jan 1961
Ammonium Hydroxide, Tech	AmmH	O-A-451E	Aug 1968
Ammonium Nitrate, Prilled	AN	MIL-A-50460(1)	July 1972
Ammonium Perchlorate	AmmPer	MIL-A-23442A(1)	Sept 1966
Ammonium Perchlorate for Solid Proplnt Grains Mark 75 and Mark 76	AmmPer	MIL-A-23946(1)	Sept 1966
Ammonium Perchlorate, Special Coarse for Solid Proplnt Grains Mark 75 and Mark 76	AmmPer	MIL-A-23948(1)	Sept 1966
Ammonium Perchlorate, Tech	AmmPer	MIL-A-192B	Sept 1965
Ammonium Picrate (Expl D)	AmmP	MIL-A-00166B	Aug 1968
Antimony Sulfide (For Use in Ammunition)	Sb_2S_3	MIL-A-159D	May 1972
Asphaltum (Gilsonite)		JAN-A-356	June 1946
Barium Carbonate (For Use in Ammunition)	$BaCO_3$	MIL-B-624A	April 1969
Barium Chromate	$BaCrO_4$	MIL-B-550A(1)	March 1967
Barium Nitrate, Technical	$Ba(NO_3)_2$	MIL-B-162D	Feb 1968
Barium Oxalate (For Use in Ammunition)	$Ba(C_2O_4)$	JAN-B-660	Aug 1948
Barium Peroxide	BaO_2	MIL-B-153A(1)	May 1969

Material	Abbreviation or Formula	Specification	Date
Barium Stearate	$Ba(C_{18}H_{35}O_2)$	JAN-B-366	July 1946
Binder, Cellulose Nitrate Camphor (For Pyrotechnic Mixtures)		MIL-B-10854	Jan 1951
Butyl Acetate, Normal (For Use in Org Coatings)	n-BuAc	TT-B-838A	Aug 1959
Butyl Acetate, Secondary (For Use in Org Coatings)	sec-BuAc	TT-B-840B	Jan 1960
Butyl Alcohol, Normal (For Use in Org Coatings)	C_4H_9OH	TT-B-846B	April 1960
Butyl Alcohol, Secondary (For Use in Org Coatings)	sec-C_4H_9OH	TT-B-848B	Dec 1959
Butyl Stearate, Normal	$C_{17}H_{35}COOC_4H_9$	MIL-B-21465	July 1958
Cadmium Oxide	CdO	MIL-C-6151A	Oct 1969
Calcium Carbonate	$CaCO_3$	MIL-C-00293A	Sept 1967
Calcium Resinate		MIL-C-20470A	Sept 1964
Calcium Silicide	$CaSi_2$	MIL-C-324B	Oct 1969
Calcium Stearate	$Ca(C_{18}H_{35}O_2)_2$	JAN-C-263	Sept 1945
Carbon Black (For Use in Explosives)	C	MIL-C-306B(2)	Jan 1968
Cellulose Acetate (For Use in Propellant Powder)	CA	MIL-C-20301	Nov 1951
Cellulose Nitrate (Pyroxylin) Rods (For Use in Ammunition)	NC	JAN-C-801	June 1949
Cellulose, Cotton (For Use in Explosives)		MIL-C-206A	Aug 1962
Cellulose, Regenerated, Strip (For Use in Ammunition)		JAN-C-677(2)	Dec 1951
Charcoal (For Use in Ammunition)	C	JAN-C-178A(1)	Oct 1949
Composition B	Comp B	MIL-C-401C(2)	Nov 1972
Composition B	Comp B	MIL-C-00401D	Feb 1969
Composition B3	Comp B3	MIL-C-45113A	Oct 1963
Composition B4	Comp B4	MIL-C-46652(1)	Dec 1962
Composition C3	Comp C3	MIL-C-427A	Sept 1968
Composition CH-6	Comp CH-6	MIL-C-21723A(1)	Nov 1972
Composition D2	Comp D2	MIL-C-18164A(1)	March 1969
Composition 86/14 RDX/Wax		MIL-C-60051	Aug 1964

Material	Abbreviation or Formula	Specification	Date
Composition, Delay		MIL-C-13739A	Nov 1965
Composition, Tracer, R-45 Igniter, K-29		MIL-C-14334	June 1956
Composition, Tungsten, Delay		MIL-C-48141	March 1973
Compositions A3 and A4	Comp A3 & Comp A4	MIL-C-440B	July 1961
Copper (99.0% Min Cu)	Cu	AMS-4740	Jan 1971
Copper, Powdered (For Use in Ammunition)	Cu	JAN-C-768	May 1949
Cupric Oxide	CuO	MIL-C-13600A	May 1962
Dextrin, Technical	$(C_6H_{10}O_5)_x$	MIL-D-3994B	Aug 1970
Diatomaceous Earth	Kieselguhr	MIL-D-20550B	Aug 1968
Di-2-Ethylhexylphthalate	$C_6H_4[COOCH_2CH(C_2H_5)C_4H_9]_2$	MIL-D-13796A	May 1962
Di-(2-Ethylhexyl) Sebacate	$(C_4H_8COOC_8H_{17})_2$	MIL-D-10692	Nov 1952
Dibutylphthalate, Technical	DBuPh	TT-D-301B	April 1960
Diethylene Glycol	DEGc	MIL-D-23296A	June 1972
Diethylphthalate (For Use in Explosives)	DEPh	JAN-D-242	July 1945
Diphenylamine, Technical	DPhA	MIL-D-98A	May 1962
Dynamite, Commercial		MIL-D-60365(1)	Nov 1968
Dynamite, Military, MI		MIL-D-45413A(3)	Feb 1966
Dynamite, NG, Gelatin		MIL-D-28606	Aug 1972
Ether, Diethyl, Technical	Eth	MIL-E-199A(1)	Dec 1966
Ether, Petroleum, Technical	Petr eth	O-E-751B	April 1956
Ethyl Alcohol (For Ordnance Use)	Alc	MIL-E-463B	May 1962
Ethyl Centralite (Carbamite)	EtCentr	MIL-E-255A(1)	June 1966
Ethylene Glycol, Technical	EGc	MIL-E-9500A(2)	Dec 1972
Explosive Composition A5	Comp A5	MIL-E-14970A(1)	Jan 1973
Explosive Composition HTA-3	HTA-3	MIL-E-46495A(1)	April 1972
Explosive Compositions, HBX Type	HBX	MIL-E-22267A	May 1973
Explosive, Plastic-Bonded Molding Powder (PBXN-5)	PBXN-5	MIL-E-81111A	Feb 1973
Graphite, Dry (For Use in Ammunition)		MIL-G-155A(2)	July 1967
HMX (For 30mm Ammunition)	HMX	MIL-H-50589	Oct 1970
HMX	HMX	MIL-H-45444A(3)	July 1962

Material	Abbreviation or Formula	Specification	Date
Hydrazine Solution (22%)	$H_2N.NH_2$	MIL-H-22251	Nov 1959
Hydrochloric Acid, Technical	HCl	O-H-765B	May 1969
Incendiary Mixture PT1		MIL-I-10223A	Aug 1950
Iron Oxide, Black, Technical	Fe_3O_4	MIL-I-275B	May 1969
Iron Oxide, Ferric, Red Dry (Natural and Synthetic)	Fe_2O_3	MIL-I-706A	Aug 1962
Lactose, Technical	$C_{12}H_{22}O_{11}.H_2O$	MIL-L-13751A	May 1966
Lead Azide	LA	MIL-L-3055A	Sept 1962
Lead Azide RD-1333	LA RD-1333	MIL-L-46225C	Aug 1968
Lead Azide, Special Purpose (For Use in Ammunition)	LA SP	MIL-L-14758	May 1968
Lead Carbonate, Basic, Dry (For Ordnance Use)	$2PbCO_3.Pb(OH)_2$	MIL-L-18618(1)	April 1956
Lead Chromate (For Use in Ammunition)	$PbCrO_5$	JAN-L-488	Aug 1947
Lead Dioxide, Technical	PbO_2	MIL-L-376B(2)	Feb 1968
Lead Mononitroresorcinate	LMNR	MIL-L-46496(1)	Aug 1962
Lead Nitrate, Technical	$Pb(NO_3)_2$	MIL-L-20549A	Jan 1968
Lead Styphnate, Basic	basic-LSt	MIL-L-16355C	March 1961
Lead Styphnate, Normal	n-LSt	MIL-L-757A	Sept 1968
Lead Thiocyanate	$Pb(SCN)_2$	MIL-L-65A	Feb 1968
Lead-Antimony (For Use in Ammunition)	Pb-Sb	MIL-L-13283B	Aug 1970
Lithium Chloride (For Use in Ammunition)	LiCl	MIL-L-14752	May 1968
Magnesium Oxide, Calcined (For Use in Ammunition)	MgO	MIL-M-14779(1)	April 1972
Magnesium Powder (For Use in Ammunition)	Mg	MIL-M-382B	July 1970
Magnesium Stearate (For Use in Ammunition)	$Mg(C_{18}H_{35}O_2)_2$	MIL-M-542A(2)	Jan 1973
Magnesium-Aluminum Alloy, Powdered	Mg-Al	JAN-M-454(1)	Feb 1952
MOX-2 Explosive	MOX-2	MIL-M-46263	Aug 1960
Mercurous Chloride (For Ordnance Use)	HgCl	JAN-N-201(1)	Feb 1971
Mercury Fulminate	MF	JAN-M-219	May 1945
Methyl Centralite (For Use in Ammunition)	MeCentr	MIL-M-19719A	July 1960
Nitric Acid, Technical	NA	O-N-350A	April 1968
Nitrocellulose	NC	MIL-N-244A	Oct 1965

Material	Abbreviation or Formula	Specification	Date
Nitroglycerin	NG	MIL-N-246B	Feb 1962
Nitroguanidine (Picrite)	NGu	MIL-N-60501C	Feb 1973
Oxamide Explosive Composition		MIL-O-60371	June 1966
Polyisobutylene (For Ordnance Use)		MIL-P-13298A	Nov 1963
Polyvinyl Acetate Aqueous Emulsion (For Use in Ammunition)	PVAAE	MIL-P-50855	March 1971
Polyvinyl Chloride (For Use in Pyrotechnics)	PVC	MIL-P-20307	Nov 1951
Potassium Dinitrohydroxy Hydrobenzofuroxan (For Use in Ammunition)	KDNBF	MIL-P-50486	March 1971
Potassium Nitrate	KN	MIL-P-156B	April 1956
Potassium Perchlorate	KPer	JAN-P-217A(1)	June 1966
Potassium Picrate	KPic	MIL-P-10830	Jan 1951
Potassium Sulfate (For Ordnance Use)	K_2SO_4	MIL-P-193A(1)	Aug 1965
Powder, Black	BkPdr	MIL-P-223B(1)	July 1963
Powder, Black, Sodium Nitrate		JAN-P-362(1)	Dec 1946
Powder, Metal, Atomized (For Use in Ammunition)		MIL-P-14067B(3)	Nov 1971
Powder, Molding, PBX 9010	PBX 9010	MIL-P-45447	July 1960
Powder, Molding, PBX 9404	PBX 9404	MIL-P-45446A	Aug 1962
Powder, Photoflash		MIL-P-466A	April 1951
Powder, Propellant, Cordite N		MIL-P-17449(2)	May 1972
Powder, Propellant, AA (For 20mm gun)		MIL-P-17646(2)	May 1972
Powder, Propellant, M10		JAN-P-715	Jan 1949
Propellant, Double Base, Type N-2	JPN	MIL-P-18617	June 1955
Quinonedioxime (For Rocket Motor, 3.0 inch, Mark 32, Mod O)	$HONC_6H_4NOH$	MIL-Q-23703(1)	Feb 1965
RDX	RDX	MIL-R-398C(1)	Oct 1963
RDX, Desensitized		MIL-R-13742(1)	April 1972
Silicone Rubber, Foaming Compound (For Use in Ammunition)		MIL-S-50457	Jan 1970
Sodium Azide, Technical	SA	MIL-S-20552A	July 1962
Sodium Carboxymethyl Cellulose (For Use in Ammunition)	NaCMC	MIL-S-51132A	Sept 1964
Sodium Nitrate	SN	MIL-S-322C	Feb 1968

Material	Abbreviation or Formula	Specification	Date
Sodium Resinate, Technical	SRes	O-S-607C	March 1967
Sodium Sulfate, Anhydrous, Technical (For Use in Ammunition)	SS	MIL-S-50004A	June 1968
Sodium Oxalate, Technical	SOx	JAN-S-210	May 1945
Strontium Nitrate, Anhydrous	$Sr(NO_3)_2$	MIL-S-20322B	April 1973
Strontium Oxalate	$SrC_2O_4.H_2O$	MIL-S-12210A	July 1956
Strontium Peroxide	SrO_2	MIL-S-612A	Sept 1964
Titanium Dioxide, Dry	TiO_2	MIL-T-48149	April 1973
Trichlorotrifluoroethane (For Use in Ammunition)	$C.Cl_2FCClF_2$	MIL-T-14757	May 1968
Tungsten Powder (For Pyrotechnics)	W	MIL-T-48140	April 1972
Wax, Hydrocarbon (For Ordnance Use)		MIL-W-13945B	June 1968
Zinc Dust (For Use in Pyrotechnics)	Zn	MIL-Z-365A	March 1968
Zinc Oxide, Technical	ZnO	MIL-Z-291E(1)	Aug 1970
Zirconium (Granular and Powdered)	Zr	MIL-Z-399D(1)	April 1973
Zirconium-Nickel Alloy, Powdered	Zr-Ni	MIL-Z-11410B	Feb 1968

NOTES:

1) The above lists unclassified Federal, Military, and Departmental Specifications
2) Revisions are indicated by a suffix letter to the basic number
3) Amendments are indicated by a suffix number in parenthesis to the basic number
4) The date listed is that of the latest issue, revision or amendment as of December 1973
5) All requests for copies of Specifications should state title and identifying number. Requests should be submitted to: Commanding Officer
   Naval Publications & Forms Center
   5801 Tabor Avenue
   ATTN: NPFC
   Philadelphia, Pennsylvania 19120
6) Federal Specifications will not generally be furnished by the Naval Publications & Forms Center to commercial concerns unless required in connection with a bid or contract, or for sufficient other justification. These documents may be purchased from:
   Business Service Center
   General Services Administration
   Washington, DC 20405

Explosives, Color Identification by. See COLOR REACTIONS AND COLOR REAGENTS in Vol 3 of Encycl, pp C405-L to C420-L

Explosives, High Density. L.F. Audrieth & D.D. Sager in USP 2482089-091(1949) & CA **44**, 839(1950) disclosed three tricomponent HE compns for liquid loading into shells, bombs etc. In the first patent Cyclotrimethylenetrinitramine is suspended in a soln of Trinitrophenylmethylnitramine in TNT. In the second patent EDNA is suspended in a solution of the same two HE's. In the third patent EDNA is suspended in a soln of PETN in TNT. In all cases solidification occurs after loading

Explosives, Improvised. Improvised explosives are those usually made at the site from readily available oxidizers, fuels, binders and stabilizers. Some of compns were cited during oral presentation by Dr H.J. Matsuguma at Explosives Division, FRL, Picatinny Arsenal, Dover, NJ. No report was issued [See also Explosives, R&D (Research & Development)]

Ref: O.E. Sheffield, Editor, Expls Div Newsletter, No **25**, p 9(July 1972)

Explosives, Introduction to. A booklet entitled: "Introduction to Explosives", Publication NBC 002, 86pp, 68 illustrations, can be obtd for $3.00 from the National Bomb Data Center, International Association of Chiefs of Police, 11 Firstfield Rd, Gaithersburg, Maryland 20760. Accdg to review given by G. Cohn, Edit, Expls & Pyrots **7**(1), 1974, the booklet is written in simple terms that provide a general understanding of the nature of explns & expls. Written to furnish a source of background info for police, fire, and security officials, it can also serve students, management, or others who would benefit from having a brief, clear, general overview but do not need a textbook or technical manual

Explosives Consumption. The apparent consumption of industrial expls in the US for 1972 was 2670 million pounds (about 1210 million kilograms). Apparent consumption of expls and blasting agents increased in all use categories except in construction and metal mining

Used In:	Consumption 1972		Change from 1971
	Million lbs	%	%
Coal Mining	1213	45.4	+13.0
Quarrying and Non-metal Mining	494	18.5	+ 0.8
Railway and Other Construction	467	17.5	– 2.3
Metal Mining	431	16.1	– 5.8
Seismograph	19	0.7	+7.7
All Other	47	1.8	+17

The top-ranking states: Ky, Pa, Indiana, West Va, and Arizona, consumed 1170 million pounds or 44– of all explosives and blasting agents used

Note: The apparent consumption of industrial expls in the US for the 5 previous years is shown below for reference. Info is from surveys like Ref 1 but of earlier years

Used In:	Consumption in Million lbs				
	1971	1970	1969	1968	1967
Coal Mining	1071	962	820	684	662
Quarrying and Non-metal Mining	490	480	439	398	409
Railway and Other Construction	478	455	441	412	412
Metal Mining	457	447	471	403	328
Seismograph	17	25	41	41	86
All Other	40	24	14	10	8

Refs: 1) Anon, "Apparent Consumption of Industrial Explosives and Blasting Agents in the United States, 1972", Mineral Industries Surveys, Explosives, Annual, July 1973
2) G. Cohn, Edit, Expls&Pyrots **7**(1), 1974

Explosives Containing Nitrostarch. Nitrostarch containing ignition mixtures are prevented from gelling prior to application to the ignition element by incorporating one or

more monohydric alcohols containing no more than six carbon atoms
Ref: W.D. Trevorrow, USP 2484131 (1949) & CA **44**, 2247 (1950)

Explosives Containing Paraffin and Gasoline. A compn useful as a propellant is derived from a mixture, with plasticized NC of dry potassium chlorate impregnated with Manila copal resin and of dry potassium chlorate/charcoal impregnated with a gasoline-paraffin distillate. Burning rate of the grains obtained by extrusion or sheeting/cutting is controlled by the amount of chlorate-resin present
Ref: S.J. McClung, USP 2483589 (1949) & CA **44**, 2246 (1950)

Explosives RD, History of. All expls named by an RD designation originated in the Explosives Research and Development Establishment (ERDE) of the Ministry of Technology in Waltham Abbey, Essex, England. ERDE, actively engaged in R&D of expl formulations for many years, found early that it was not enough to label a compd merely by its chem formula. Form of the ingredients, method of manuf, as well as several other factors affect the final product. Thus RD 1333 Lead Azide (LA) is a very specific expl that differs significantly from other LA's. Another example is a new form of dextrinated LA called RD 1352. Both expls are in the 1300 series, the block for primary expls

Accdg to Dr John M. Jenkins, the ERDE's most known expl, Cyclonite, does not have a number but was merely labeled with the letter X, making it RDX. Legend has it that years ago a researcher forgot the number of a particular kind of Cyclonite. The information was locked in a safe, probably for security reasons. But when an immediate designation was required, the researcher suggested calling it X. It is not guaranteed that this legend is true, but it is one version of the nomenclature of RDX
Ref: G. Cohn, Edit, Expls&Pyrots **7**(1), 1974 (Review)

Explosives Sensitivity Data. Card-gap and projectile sensitivity data are presented by Watson (Ref 1) for a wide variety of expl compns tested at the USBurMines laboratories in more or less standard test geometries. The results of both tests are in good agreement in that they provide the same sensitivity ordering for different subclasses of expls. Least sensitive were homogeneous liquids that did not exhibit a tendency to undergo low-velocity detonation, AN-FO (Ammonium Nitrate-Fuel Oil), and most cast military expls. Of intermediate sensitivity were pressed and powdered military expls, cast Pentolite, permissible and nonpermissible water-based expls, and one commercial two-component expl. The most sensitive were permissible and nonpermissible Dynamites and expls susceptible to low-velocity detonations
Refs: 1) R.W. Watson, "Card-Gap and Projectile Impact Sensitivity Measurements, A Compilation", USBurMines Information Circular **IC 8605** (1973)
2) G. Cohn, Edit, Expls&Pyrots **7**(1), 1974 (Review)

Explosive Trains. An expl train is an assembly of elements arranged in order of decreasing sensitivity. The function of the train is to accomplish controlled augmentation of a small impulse into one of suitable energy to cause the main chge of the munition to function

Expl trains may be divided into two general classes: *high explosive trains* and *low explosive trains*, according to the type of expl used in the main chge. An expl train may also be designated accdg to the item in which it is assembled. The most common expl train is the *fuze explosive train.* If a bursting chge is added, it is commonly called *bursting charge explosive train.* An example of a low expl train is the *propelling charge explosive train*

Essential elements of a high expl train are:
a) a primary or low expl chge contained in a suitable housing
b) an intermediate chge of primary expl (most commonly Lead Azide) in which transition from burning to detonation takes place
c) a secondary high expl chge (RDX) that intensifies the shock output from the intermediate chge

d) a main chge consisting of a secondary HE (TNT) that produces the desired effect
e) auxiliary elements for special purposes are leads & relays, delay or time elements, or a booster chge

The effectiveness of one chge in initiating another is determined by the props of the expl, its loading density, and the dimensions & confinement of the chge. The effective output of a donor charge increases systematically with its diameter. The optimum diameter of an acceptor, from the point of view of the air gap across which it can be initiated, is slightly less than the diam of the donor, especially for well confined columns of expls

See also Section 9 "Physical Tests for Determining Explosive and Other Properties of Detonators, Primers, Igniters and Fuzes" in Vol 4 of Encycl, pp D1078ff

Refs: 1) Anon, "Ordnance Explosive Train Designers' Handbook", NOLTR 1111(1952), Chapters 1 & 2 2) Anon, "Explosive Trains", AMCP 706-179(1965), Chapters 1, 2 & 3

Explosivo núm 3. Spanish permissible expl listed in Vol 3 of Encycl, p C455-L and in Vivas, Feigenspan & Ladreda, Vol 2(1946), p 379

Explosivos ácidos. Span expls consisting of mixts of concd nitric acid with aromatic nitrocompds, such as MNB, MNT, DNB, DNT, etc. Egs: a) HNO_3 71.9 & MNB 28.1% and b) HNO_3 60 & DNB 40%. They are actually Sprengel type expls

Ref: E.E. Sancho, "Química de los Explosivos", A. Aguado, Madrid (1941)

Explosivos antigrisú. Span permissible expls, listed as Antigrisú (Explosivos) under Coal Mining Explosives, Permissible in Vol 3, p C454-R

Explosivos F.E., such as F.E.-1, F.E.-2 and F.E.-3, are Span nonpermissible expls listed in Vol 3, p C442-L and in Vivas, Feigenspan & Ladreda 2(1946), 384

Explosivos de seguridad. Span permissible expls. One of such expls, known as *Nitramita* was manufd at the Fábrica de Granada. It contd AN 80 & TNT ("trilita" in Span) 20%. The Nitramita manufd at the Fábrica de dinamita de Galdácano contd AN 88 & DN-Naphthalene 12%

Ref: Vivas, Feigenspan & Ladreda 2(1946), 378

Explosivos de seguridad de la Fábrica de dinamita de Galdácano, listed on p 378 of Vivas, Feigenspan & Ladreda 2(1946) also listed in Vol 3 of Encycl, p C455-L, under Coal Mining Explosives, Permissible

Explosophore. See under Auxoexplose or Auxoplosophore; Explosophore or Plosophore in Vol 1 of Encycl, pp A513-R to A514-R

EXPLOTRON. A light generator designed to extract, for useful purposes, the brilliant flashes produced by explosives has been patented by De Ment (Refs 1–9). The device called an "explotron" takes an expln at short range and hitherto inaccessible ranges and plucks the pristine light from crushing blast, flying debris, and deadly nuclear radiation & radioactive material that may be present. The principles upon which the explotron is based are the enormous velocity difference between light (186000 miles/sec) and blast (several miles /sec) and the fact that light can be cleanly deflected in ways that blast cannot

In essence, the explotron is a ducted structure optically coupled at one angle to a frangible optical member which is reflective to electromagnetic radiation and destructible by non-electromagnetic radiation. Upon actuation of the expl light source, the electromagnetic radiation is directed by a mirror to the irradiable target, and the slower non-electromagnetic radiation breaks the mirror and moves along the passage into a chamber where the blast & debris are diverted from the target

There are three basic varieties of explotrons: a) furcated or branched b) rectilineal or retroreflective and c) hybrids of a & b

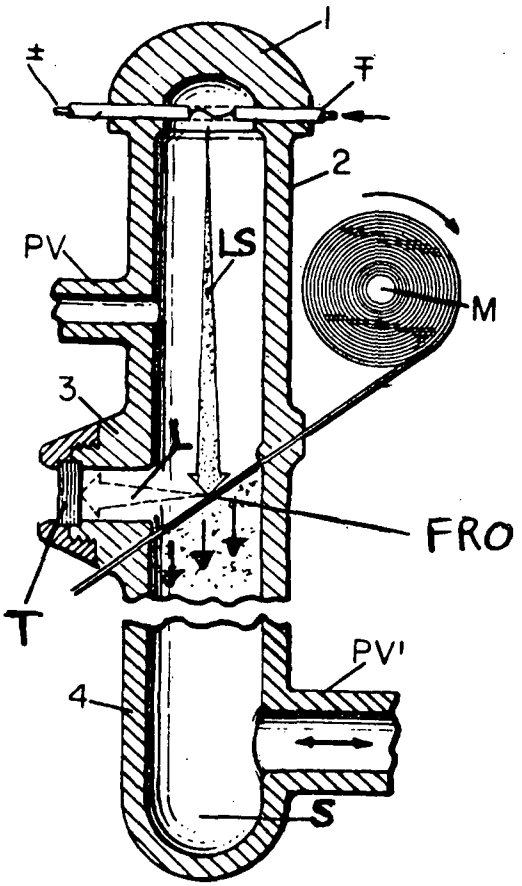


FIG 1
FURCATED OR "T" CONFIGURATION EXPLOTRON

A furcated explotron (Fig 1) consists of a main tube with at least one side-arm angularly conjoining the main tube. At one end of the main tube a light generating expl (optoexplosive) is sited & fired. In Fig 1 the expl light source is contained within **1**, the light generating leg is **2**, **LS** is the light-shock admixture; light **L** first strikes the frangible reflective optic **(FRO)**, fed off roll **M** into the juncture, and into the side arm **3** that is coupled with irradiable target **T**; slower-running shock & blast and debris **S** break out **FRO** and sink into leg **4**; PV & PV′ may be side ducts for special gas loading, for vacuumization or for explosion plasma manipulation

A branched or furcated explotron may be variously configured to increase the light-from-blast separation efficiency and to optimize the coupling of the reflected radiation with the irradiable target **T**. Fig 2 shows the "Y" Configuration Explotron. Both "T" & "Y" forms of the explotron are considered unitary or modules which can be expanded into zig-zig, blind-alley, and like forms. Each may be optoexplosively and blast fluidically designed according to need

The rectilineal or retroreflective explotron is simply a straight pipe or tunnel. At the upstream end there is sited the irradiable target **T** which, depending upon the power-

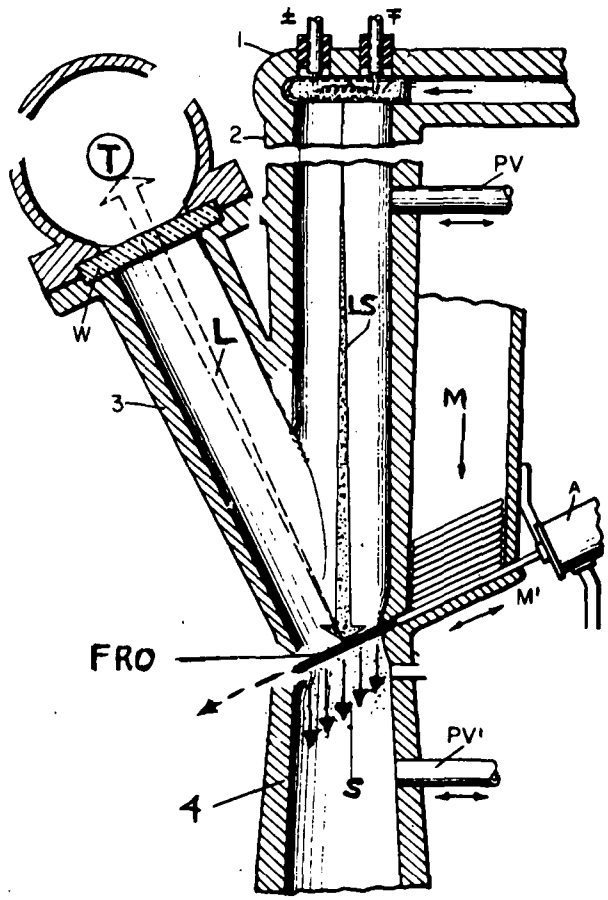


FIG 2

ing of the system, the duct, ancillary venting means, type of optoexplosive light generator, and longitudinal profile, may or may not be hardened to take care of shock & pressure below the point of destruction by indirect explosion impact. Downstream from T is the optoexplosive energy source (male or female configuration, Figs not shown). The **FRO** (frangible reflective optic) is a plane, convex or concave mirror lens mounted 90° to the long axis of the system. The T may be either male or female to match the optical requirements. As with the furcated explotron, the rectilineal explotron may incorporate a variety of light-manipulating & kinetic energy-degrading components. These range from flux redistributors, to simple high albedo duct wall surfacings, to ablatives and turbulencing vanes, to low-pressure venting pipes

Hybridized or multimodular explotrons are combinations of the furcated & rectilineal types. Each type can be considered building blocks for more complex systems. The purpose of compounding the basic elements in each type of explotron is foremost the defeating of material energies, especially hot plasma close-in to a soft or semihardened target. Also, there is a range of choices of optoexplosive devices which, otherwise, emit inordinate amts of kinetic energy; and where pressure differentials are to be made within the system; or for speciality gas loadings, liquid loadings, or when parts of the system are extended into a body of water. In hybridizing a branched system, such as a T explotron (Fig 1), with a rectilineal one (Fig, not shown), the latter can be emplaced in the side-arm of the former. Between the angled frangible reflective optic (FRO) of the furcated explotron and the FRO of the rectilineal unit the target, T′, is sited; the rear end of the T is hardened, and the face of the T receives the now upstream retroreflected optoexplosion light

There was also developed & patented by De Ment (Ref 10) a tellurian (underground) explotron for close-in treatment of an irradiable target by explosion-formed electromagnetic energies characterized as reflectable off a frangible reflective optic such that the target is not overwhelmed by non-reflectable expln energies like blast & plasma. A typical tellurian explotron (shown in Fig 3) is a main duct horizontally sited below ground, optic (FRO) within one end of which is positioned and fired a mass of chemical or nuclear expl that generates electromagnetic & kinetic energies (black arrows). Junctured midway or at any other convenient site along the main duct is at least one ductoid branch member, which rises to the ground surface and couples with the irradiable target. At the furcation of main duct & branch there is emplaced at a suitable angle a FRO, such as an aluminum foil mirror, which delivers the faster moving, reflectable radiation (white arrows) via the branch to the target. The slower-moving non-reflectable energies transit the main duct and break out the FRO and deposit in the ends of the main tunnel (small arrows)

Dr DeMent tested the explotron by detonating a high explosive in an abandoned mine shaft on Fleming Island, Alaska in 1967 and succeeded in getting a 20-sq ft light beam off an Al foil mirror. Expls traditionally have been military & industrial "work-horses" because of their blast energy, but tremendous amts of light also are released by expls. This light energy is usually wasted and in most cases considered an undesirable product. Up to 80% of the energy of high-altitude nuclear explns can be released as wasteful radiant energy. Not all of the energy in an expl goes out as radiant energy but a chemical or nuclear expln can be optimized for best light output. Electrical energy often goes thru a series of heat-wasting steps before it becomes intense light

There are many useful applications for clean explosion light. Several of these already have been explored with current light sources. Physics, chemistry, metallurgy, and life sciences use intense or special wavelength light for the irradiation of many objects from spacecraft to living tissue and

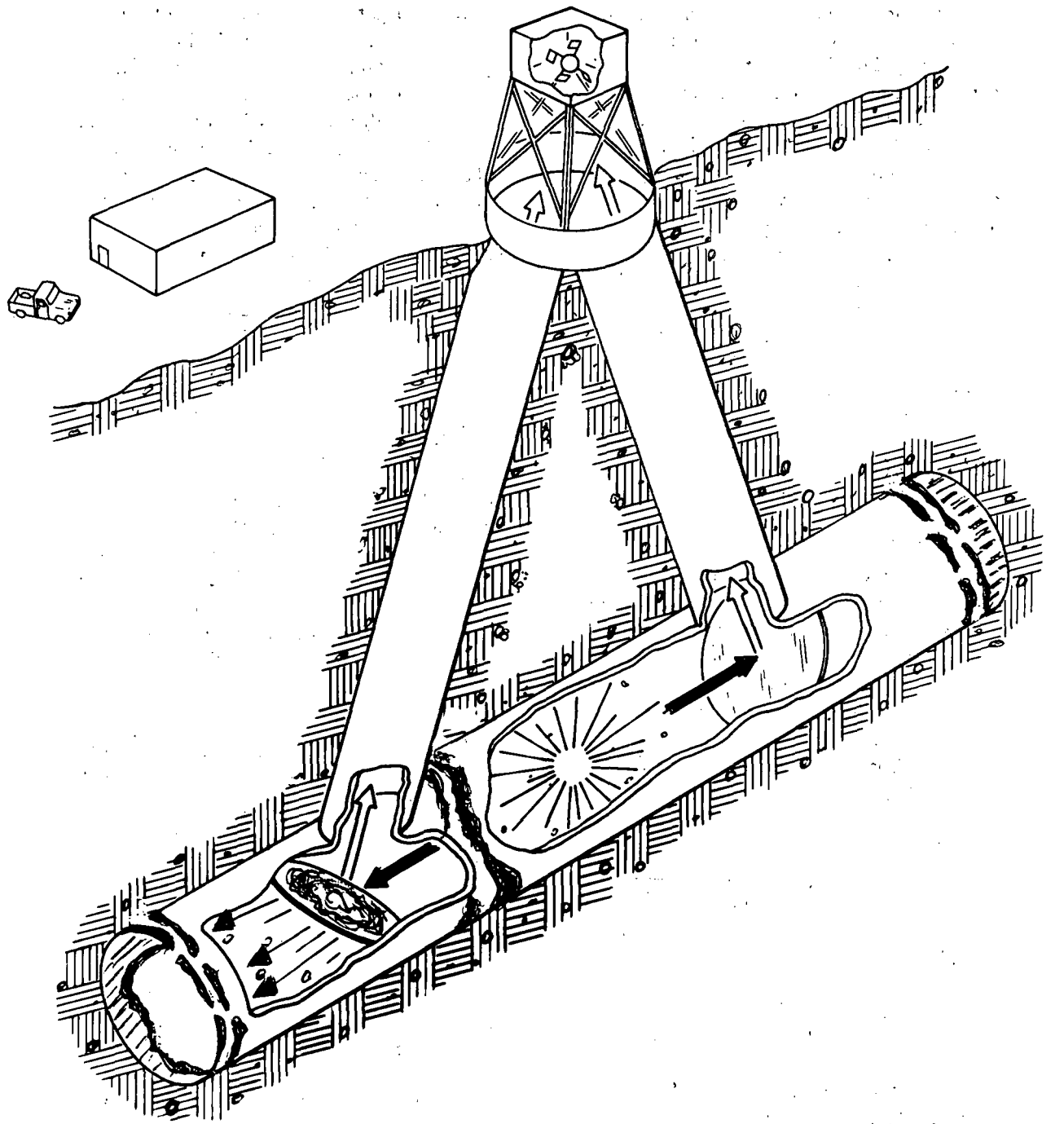


FIG 3
TELLURIAN (UNDERGROUND) EXPLOTRON

from the effects of sunlight in space to eye & brain surgery. Superintense light which appears to be neatly & efficiently extracted by the explotron could be employed for making new alloys, glasses, ceramics, and unusual high-temperature chemicals. It also might find application in the study of flash burn & diseased tissue research. DeMent does not restrict his projected applications to those requiring visible light. Depending on the selection of explns (chemical, electrical or nuclear) the wavelength spectrum can include soft X-ray, ultraviolet, visible, infrared, and even the transition region bordering microwaves. DeMent feels the explotron opens the door to a new technology of special devices that produce large amts of useful radiant energy

See Refs for addnl details

Refs: 1) S.V. Jones, "Device Seeks to Harness Energy from Explosions", NY Times, p C59 (30 Nov 1968) 2) J. DeMent, "Explodable Light Source and Laser Light Generator", USP 3414838 (3 Dec 1968) 3) Staff, "A Concept, Optoexplosives, and Its Resultant Device, the Explotron", C&EN **46** (53), 55 (16 Dec 1968) 4) Staff, "Useful Light is Split from Blast Explosion", ProductEngrg **40**(2), 21 (27 Jan 1969) 5) Staff, "Capturing Bright Light from High Explosives", Optical Spectra **3**(1), 22–23 (Jan–Feb 1969) 6) Staff, "Optoexplosion Principle and 'Explotron' Separate Light from Blast", MachDesign **41**(3), 12 (6 Feb 1969) 7) A.F. Plant, "Lightning without Thunder"} Industrial Res **11**(6), 62–64 (June 1969) 8) Staff, Möglische Anwendung der hohen Lichtenenergie von Explosionen", Laser u Strahlentechnik (Munich) **1**(2), 61 (June 1969) 9) J. DeMent, "Method of Separating Light Energy from Shock Wave Energy", BritP 1159144(23 July 1969)l USP 3537031 (27 Oct 1970); and USP 3546623 (8 Dec 1970) 10) J. DeMent, "Tellurian Optoexplosive System Including Nuclear Explosive Light Generator and Target Irradiation", USP 3715596 (6 Feb 1973)

Exponents of a Polytropic Curve of Explosion Products of Condensed Explosives. Accdg to Apin et al(Ref 1), the adiabatic curve of expln products at the front of a detonation wave may be described by the polytropic law: $p=Av^{-n}$, where p=pressure, A=function of entropy, v=volume of expln products and n=polytropic exponent (See Ref 2, pp D290-R & D474-R). The exponent n (also designated as γ) depends mainly on the composition. The influence of temperature and pressure may be neglected. Experiments performed with expl compds and mixtures showed that over a wide range of temps and pressures of detonation, the exponent (n) of the polytropic curve of expln products may be obtd from the values of exponents of the individual products: $n^{-1}=\Sigma\beta_i n_i$, where β_i=mole part of a given expln product and the values of n_i are the following: $n_{H_2O}=1.9$, $n_{N_2}=3.7$, $n_{CO}=2.85$, $n_C=3.55$, $n_{CO_2}=4.5$ and $n_{O_2}=2.45$ (Ref 1)

The work of Apin et al on calculation of exponents of a polytropic curve of expln products of condensed expls was summarized in Ref 2, pp D474-R & D475-L

Refs: 1) A.Ya. Apin et al, ZhPriklMekh i TekhnFiz **1961**, No 5, 117–18 & CA **56**, 11871(1962) 2) Encycl of Expls, PATR **2700**, Vol **4**(1969), pp D290-R and D474-R & D475-L

Expression is the separation of liquid from a two-phase solid-liquid system by compression of the system under conditions that permit the liquid to escape, while the solid is retained between the compressing surfaces. Expression serves the same purposes as filtration but is distinguished from the latter in that the pressure is applied by movement of the retaining walls instead of pumping the material into a fixed space. Expression is usually employed to separate systems that are not easily pumped. It is also used instead of filtration when a more thorough removal of liquid from the cake is desired. The usual equipment for expression is a hydraulic press. Most of the common vegetable oils are produced by expression, In the expl in-

dustry, expression is used in the manufacture of propellants, namely during dehydration of a partly colloided block of NC. This operation is described in Vol 3 of Encycl on p C399, under Colloiding Agents and Colloidal Propellants

Refs: 1) J.H. Perry Edit, "Chemical Engineers' Handbook", McGraw-Hill, NY (1950), pp 1073–75, F. Gurnham, "Expression" and in 4th Edition (1963) **19**, 39–41 2) R.E. Kirk & D.F. Othmer, Edits, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **6** (1951), p 158, under Fats (See also under Cottonseed and Soybeans) 3) Ibid, 2nd Edition, Vol **14** (1967), p 183, under Essential Oils

Exsudation. Fr for Exudation

Exsudation par étuvage. A French test described in Vol 1 of Encycl, p XI, under Exudation (or Sweating) Tests

Extending (Stretching or Lengthening) of Gelatin-Dynamites. Under this term are known Dynamites contg less than 20–25% NG–NC jelly and still able to be worked-up to a plastic mass which could be packed in the usual cartridge machines. Such expls were developed during WWI in Germany when a shortage of NG or of other "explosive oils" became acute. The extending (stretching) was achieved by replacing part of NG–NC jelly with aromatic nitrocompounds, miscible with NG–NC. Some aromatic hydrocarbons, such as naphthalene also could be incorporated. An oxidizing material as heavy as possible, such as K perchlorate, is of advantage in such Gelatin-Dynamites, in order to obtain high plasticity and high brisance. In the case of stretched Dynamites which have a nearly complete combustn equation and acceptable fumes on expln, the above added substances do not act favorably on the permissibility, since the temperature of expln is raised considerably. In order to lower this temp, K chloride was incorporated in Germany. When other European countries adopted extended Dynamites, they started to use NaCl in permissible expls, but in England they preferred to use Amm oxalate in their "permitted" expls

Besides substances miscible with NG–NC, Germans used in some of their stretched Dynamites liquids insoluble in NG but capable, on being emulsified with it, of increasing the plasticity of the expl in the manner desired. One of such additives was developed at the Carbonitfabrik under the name of **Melan.** It was a jelly obtd by boiling glycerin with an aqueous soln of glue, for increasing the plasticity. The Dynamit AG, formerly Nobel & Co, incorporated a substance wholly or partly sol in water or which swelled up with it, such as dextrin, starch, potato flour or gums

Also a pure glycerin and so-called **Gum-Sugar** (a syrupy concd sugar soln which would not crystallize) was used. The small amts of vegetable oils, or paraffin oil and the so-called *concentrated waste sulfite liquor*, patented in Germany after WWI, also were found suitable for stretching Gelatins

In contrast to the above substances, which are carbonaceous materials and consequently require oxygen for their combstn, and raise the temp of expln, the Dynamit AG developed in 1919 the use of accurately measured quantities of satd Ca nitrate soln, which is in itself an oxidizing agent. It imparts to the expl a high degree of plasticity without affecting the sensitiveness by the attendant water content. It allows the manuf of gelatinous expls of low NG content which cartridge will have an oxygen excess and high charge limits. These expls were known as Wetter-Nobelits

Although extended or stretched Dynamites could be called Substitute Explosives (Ersatzsprengstoffe, in Ger), they should not be confused with Ersatzsprengstoffe of WWII listed in PATR **2510** (1958), pp Ger 43 & Ger 44 and also in Vol **5** of Encycl, p E122, Table E15

In Vol **5** of Encycl under DYNAMITE AND SUBSTITUTES, pp D1584-L ff are also included expls which contain no NG, nor other

expl oil. Such substitute expls are AN–FO (Ammonium Nitrate–Fuel Oil)

Another example of Dynamite-type expls contg no NG are Nitrostarch Explosives developed at the Trojan Powder Co (now called Commercial Solvents-Trojan Powder at Allentown, Pa). See Vol 5, p D1605-L

Ref: Ph. Naoúm, "Nitroglycerine and Nitroglycerine Explosives", translated from German by E.M. Symmes, The Williams & Wilkins Co, Baltimore, Md (1928), pp 333, 405, 406 & 408

Extensometer for Testing High Explosive Materials. Proposed is a design for an extensometer to measure unit length changes upon compression or tension in ¼ x 1-inch lengths of HE used for the instantaneous assembly of the component fissionable materials in nuclear devices. The length change sensors are resistance foil strain gages cemented to opposite sides of the center of a 12 x 375-mil cross-sectional area in the center of a piece of spring steel 20 x 750 x 1125-mil. On the same side of the latter, at opposite ends, are soldered 1/8 x 1/2 x 1-inch brass blocks, each drilled to hold a pair of steel needles at 90° angle. Both gages are wired to one of the brass blocks and are connected to adjacent arms of a bridge for temperature compensation and to give a double signal output. A 3/4-inch coil tension spring attached to each block is used to clamp onto the specimen. The extensometer is calibrated by attaching to the dummy specimen of a Baldwin portable extensometer comparator which will cause and read extensions of as little as 10^{-5}mil

Ref: L.H. Reynolds, ASTM Bulletin No 234, 65–66 (1958)

Extent of Propagation of Detonation (or Explosion) and Tests. See "Ability to Propagate Detonation- Transmission of Detonation or Extent of Propagation of Explosion", in Vol 1 of Encycl, p VII

More detailed discussion, with numerous refs, is given in Vol 4, pp D402-L to D417-R, under "Detonation (and Explosion), Initiation (Birth) and Propagation (Growth or Spread) in Explosive Substances" and on pp D497-L to D498-R under "Detonation (and Explosion), Propagation Through Explosive Charges"

Exterior (or External) Ballistics. See under "BALLISTICS" in Vol 2, pp B7-L to B8-L

Extinction of Detonation. See under "Detonation: Attenuation, Break, Cessation, Cutoff, Decay, Dying-out, Extinction, Fadeout and Failure" in Vol 4, pp D223-R to D225-R

Extinction (or Elimination) of Muzzle Flash. See under Flash-Reducing Agents or Anti-flash Agents

Extinguishers for Incendiaries. There has been patented a process for extinguishing fires, especially those of hydrocarbons (whether gelatinized or not). The process consists of using the action of an extinguishing agent, such as a chlorinated solvent & water, spread over the fire in the form of an emulsion. It can include separately or in combination, the following characteristics:

a) the emulsion of the two products is made at the time it is utilized by means of any type of mixing
b) emulsion is assured by the presence of an emulsifying product contained in the water or in the extinguishing agent, or from a combination of two substances contained one in water and the other in the extinguishing agent
c) the extinguishing agent is carbon tetrachloride
d) the proportion of the extinguishing agent in the emulsion is ca 20%

Ref: Dubrisay & Ficheroulle, MP 38, 449–51 (1956) (FrP 1099973, 14 Sept 1955)

Extraction and Distribution or Partition. *Extraction* is the act of transferring a material

from one or more liquid or solid phases in which it is dissolved or dispersed, to another phase which is liquid. Laboratory extractions are usually made in a Soxhlet or Wiley apparatus

One of the most important laws of extraction is the *distribution (partition)* law formulated by W. Nernst in 1891: "A solute dissolved in one phase in equilibrium with another, immiscible phase, will distribute itself between the two phases so that the ratio of the concentrations in the two phases is a constant at a fixed temperature". If C_1 and C_2 are concentrations in the lighter and heavier phase, respectively, then $C_1/C_2 = k$, where k is the *distribution constant*, or the *partition coefficient*. It should be noted that this equation holds only for the simplest case, in which the molecules in each of the phases are in the same state of aggregation

Extraction has long been used for preliminary separation of mixtures into groups. With present-day techniques, it can be further used to separate and quantitatively estimate the components of mixtures of closely related substances, such as members of a homogeneous series. The basis for the first purpose is frequently that of solubility or of widely differing partition ratios. As the purpose of the 1st method is the removal of one or several components of a system, the process may be called *Extraction for Removal Purposes*

The basis for the second purpose is the use of a two-phase distribution in conjunction with a countercurrent process of some sort, so that separation may be made in spite of closely related partition ratios. This method may be called *Extraction for Fractionation Purposes*

Extraction procedure is used extensively in Explosives Laboratories and Plants (See next item)

Refs: 1) J.H. Perry, Edit, "Chemical Engineers' Handbook", McGraw-Hill, NY (1950), pp 713–753, J.C. Elgin & R. Wynkoop, "Solvent Extraction" and in 4th Edition (1963), **14**, 40–69; **16**, 3; **21**, 10–35 2) A. Weissberger, "Physical Methods of Organic Chemistry", Interscience, NY, Vol **3**(1950), pp 171–311, L.C. Craig & D. Craig, "Extraction and Distribution", with about 200 refs 3) R.E. Kirk & D.F. Othmer, Edits, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **6**(1951), pp 91–122, F. Lerman, "Liquid-Solid Extraction (10 refs) and pp 122–140, E.G. Scheibel & A.J. Frey, "Liquid-Liquid Extraction (37 refs). New Edition, Vol **8**(1965), pp 719–62, E.G. Scheibel, "Liquid-Liquid Extraction (84 refs) and pp 761–775, E.G. Scheibel, "Liquid-Solid Extraction" (17 refs) 4) T.K. Sherwood & R.L. Pigford, "Absorption and Extraction", McGraw-Hill, NY (1952), pp 391–454 (Numerous refs) 5) L. Alders, "Liquid-Liquid Extractions, Theory and Laboratory Experiments", Elsevier, Houston (1955)

Extractions Used in Explosives Laboratories and Plants. Many uses of extraction process are known, most of them dealing with liquid-solid extractions. There are, however, some liquid-liquid extractions, as, for example, extraction by ether of NB (Nitrobodies), as dissolved Nitrocompds of TNT in Spent or Waste Acids. The method described in detail in Ref 1, Chap 1, pp 21–22 (which is now out of print) is as follows: Shake the bottle with the acid contg NB vigorously to mix the contents and pour quickly 50.00g into a small tared Pyrex or porcelain dish. Transfer quantitatively the sample into about 200ml distd water placed in 400-ml beaker, cooled in ice-water. Transfer the cold soln into a 500-ml separatory funnel and add 50ml of ether. Remove the funnel from the stand and hold the stopcock firmly with the right hand. Close the top of the funnel with a ground glass stopper and, while pressing against it with the palm of the left hand, slowly invert the funnel. Immediately after this, open the stopcock to allow the vapors of ether to escape. Close the stopcock and, while holding the funnel in inverted position, mix ether with acid by gently swirling the funnel. Then open the stopcock to allow the vapors of ether (but none of the liquid) to escape. Repeat the above operations 2–3 times and then invert the funnel to normal position. Allow to stand until two layers separate. Open the stopcock

and draw off the acid-water layer into the above 400-ml beaker. Allow a few drops of ether to enter the beaker to avoid leaving any acid in the funnel. Draw off the ether layer from the funnel into the above tared dish (previously dried) and allow the ether to evaporate. For a more complete extraction repeat all the above operations using another 50-ml portion and finally a 25-ml portion of ether. Weigh the residue in the dish after evaporation and calculate as follows:

$$\%NB = \frac{(\text{Wt of Residue}) \times 100}{\text{Wt of Sample}}$$

In liquid-solid extractions, if the solid is powdery, grained or in small crystalline form, the simplest method is to pack the sample in a Gooch or sintered glass crucible inserted in the neck of vacuum flask and to treat the solid with several portions of an appropriate solvent (such as benzene, water, ether, xylene, etc). Such method is described in Ref 2, p A164-R for laboratory separation of AN from TNT in Amatol. Instead of using the crucible, a centrifuge can be employed. Similar method can be used for removal of impurities from crude solid expls, such as of TNT. On a plant scale, a large Nutsch or centrifuge can be used. Dr I.A. Krageroff, together with G.D. Clift and B.T. Fedoroff purified in 1941 at the small plant near Penns Grove, New Jersey many tons of low-grade, exudable TNT, which was melted from WWI ammunition into wooden boxes to be stored for more than 20 years at the bottom of the Delaware river. This TNT was remelted, grained, packed into Nutsches and purified by extraction with xylene, the only solvent not in short supply at that time. The laboratory method of purification of this TNT, including the graining procedure, was described in detail in Ref 1, Chap VII, where Fig 19 on p 4 showed the funnel used in the laboratory. Many tons of Grade I TNT were produced from impure TNT by Dr Krageroff's method and shipped to Europe. This was at the time when no TNT plants existed in the US. When the supply of old TNT was exhausted, Dr Krageroff constructed a small plant at Glen Wilton, Virginia. This plant, constructed in summer of 1941, was about 6 months ahead of large Govt financed Ordnance Plants. The plant at Glen Wilton exploded in winter 1941–42 after it was acquired by the Hercules Powder Co

A method similar to purification conducted at Penns Grove was used by Dr Krageroff at Keystone Ordnance Works, Meadville, Pa, but here instead of xylene, "Sellite" (aqueous soln of Na sulfite) was used for extraction of impurities

If the material cannot be extracted rapidly, it is more advantageous to use devices called "extractors". For example, extraction of NC from Cordites can be done by using a modified Wiley extractor. The method is described in Ref 3, p C539-R & C540-L. Ether-extractable material can be removed from Blasting Explosives, including Dynamites, by means of a Wiley-Richardson extractor as described in Ref 4, p D1628. The same type of extractor can be used for removal of water and water-alcohol soluble constituents (Ref 4, p D1632), for acid-soluble constituents (Ref 4, p D1634-R), or carbon tetrachloride and in acetone soluble constituents (Ref 4, p D1637)

In every analysis of propellants an extraction must be performed. The latest description of the method used by the US Armed Forces is of 1971 (Ref 5). Roweg, Soxhlet or equivalent extractor must be used and as extracting solvent, diethyl ether or anhydrous methylene chloride. Proceed as follows: Transfer an accurately weighed sample of propInt, about 5g, to the thimble of the extractor and add prescribed solvent to the tared extraction flask. Insert the open lower part of the extractor into the neck of the extraction flask and insert thru the upper opening of the extractor the bottom opening of the reflux condenser. Assemble the apparatus on the hot plate, and adjust the temperature so that the solvent drips from the condenser at the rate of 2–3 drops per second. Extract for a time which experience or preliminary work has shown to be adequate for the type of sample. It takes 3 to 20 hours and for Roweg extractor 2–3 times faster than for Soxhlet. In order to determine the completion of the extraction, allow part of the extract to drip

from the bottom of condenser into a small tared dish. Then weigh it and if no increase in wt is observed, test the residue colorimetrically. If no change in color is observed, stop extraction, disconnect the flask and evaporate the solvent, using a stream of dry air. Leave the flask overnight in a vacuum desiccator and then weigh it to the nearest 0.1mg. Return the flask to vacuum desiccator for an additional 2 hours and reweigh. If the 2nd wt is within 0.0025g of the 1st, use the lowest of these wts. If, however, the variation betw wts is more than 0.0025g, redesiccate the flask to obtain a difference of less than 0.0025g

$$\% \text{ Extractables} = \frac{A - B}{W} \times 100$$

where:

A = Wt of flask with residue, in g
B = Wt of empty flask, in g
W = Wt of sample corrected for total volatiles

% Solvent Insolubles = 100 − % Extractables
Refs: 1) G.D. Clift & B.T. Fedoroff, "A Manual for Explosives Laboratories", Lefax, Philadelphia, Vol 1(1942), Chap I, pp 21–22 and Chap VII, pp 1–5 2) Encycl of Explosives, PATR **2700**, Vol 1(1960), p A164-R 3) Ditto, Vol 3(1966), pp C539-R to C540-L 4) Ditto, Vol 5(1972), pp D1628, D1632 and D1637 5) Anon, "Military Standard. Propellants, Solid: Sampling, Examination and Testing", **MIL-STD-286B**, 30 June 1971, Method 104.1.3 "Solvent Extractive Method"

Extra Carbonit (Brit & Ger). An older mining expl: NG 35, NC 0.3, K nitrate 25.5, Ba nitrate 4.0, tan meal 34.7 & Na carbonate 0.5%
Refs: 1) Barnett (1919), 194 2) PATR **2510**(1958), p Ger 45

Extra-Dynamite of Nobel (Swed). It was patented in 1879 by Alfred Nobel and is actually *Ammongelatine Dynamit.* Its compn is given in Vol 1 of Encycl, p A295-R and in Vol 5 of Encycl, p D1604-L, under Class V Dynamites. Extra-Dynamites were also described in the following Refs
Refs: 1) Daniel (1902), 293 2) Naoúm, NG (1928), 11 3) Davis (1943), 335 4) Cook (1958), 10

"Extra" Dynamites (Amer). A trademark registered in the US Patent Office, owned exclusively by E.I. duPont de Nemours & Co (Inc) for two series of high weight strength, variable density Ammonia Dynamites. These Dynamites are NG sensitized AN Explosives with a constant weight strength of approx 65% throughout the series. In 1966 there were 8 Grades in High-Velocity and 7 Grades in Low-Velocity. A more detailed description of their properties, without revealing their compns is given in Refs 1 & 2
Refs: 1) Blasters' Hdb (1966), 34–6 and Tables 4-1 & 4-2 2) Encycl of Expls, PATR **2700**, Vol 5 (1972), p D1604-L

"Extra" Dynamites "Red Cross" of DuPont & Co. They are NG-sensitized *Ammonia Dynamites* of 20 to 60% grade strengths and a uniform density of 110, 1¼ x 8-inch, cartridges per 50 pounds. More detailed description of their properties without revealing their compositions is given in Ref
Ref: Blasters' Hdb (1966), pp 33–4 and in Table 4-1

Extralite. Cundill (Ref 1) gives composition in parts: Zn chloride 50, AN 50, solid hydrocarbons 10, liquid hydrocarbons 5 & Amm carbonate 5. In Daniel (Ref 2) its compn is given in percentages as: Zn chloride 41.37, AN 41.97, solid hydrocarbons 4.16, liquid hydrocarbons 8.34 & Amm carbonate 4.16%
Refs: 1) Cundill (1889) in its French translation, MP **5**, 333(1892) 2) Daniel (1902), 294

Extraneous Electricity, Hazards of. See ELECTRICITY, EXTRANEOUS AND HAZARDS ASSOCIATED WITH IT in Vol 5 of Encycl, pp E35–R to E55-L

Extra Powder. A variety of Giant Powder: NG 40, Na & K nitrates 40, rosin 6, sulfur 6 & kieselguhr 8%, from which it differs by containing AN coated with vaseline
Ref: Daniel (1902), 293 (Extra Powder) & 324 [Géante(Poudre)]

Extruders are devices for loading by extrusion (qv) metals, plastics, expls and pyrotechnic compositions. For example, expls such as some Amatols, Explosive D, Compositions A & C, etc which cannot be cast-loaded can be loaded by pressing them thru a steel tube by means of a worm screw working inside. An extruder resembles devices used for loading Gelatin Dynamites in paper cartridges. Such devices are known as "sausage machines". An Improved Type is shown in Ref 1, p 306, reproduced here as Fig 4

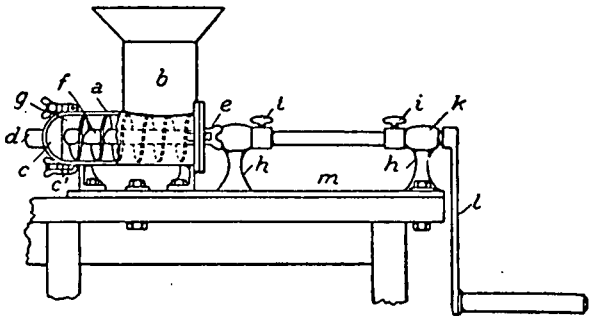


FIG 4 IMPROVED SAUSAGE MACHINE

Another type of extruding press is the so-called "macaroni press", used for making strands of smokeless propellants. Its use is described under "Extrusion" (Fig 5)

An extrusion press used in the manuf of fuel binder composite propellants for use in US aircraft jatos is shown in Fig 6

Mixing of the crystalline ingredients of oxidant, fuel & elastomer binder (butadiene-2-methyl-5-vinyl-pyridine copolymer plasticized with the formal of diethylene glycol monobutyl ether) 26% by vol is done in a sigma blade mixer. The completed mix is blocked in a hydraulic press to form a chge suitable for extrusion. The extruded strand is cut into grain blanks which are cured at elevated temp to complete the polymerization process. The cured blanks are than finished to final dimensions (Ref 3)

Addnl extruders for other applications are described in Ref 2. See also Extrusion which follows
Refs: 1) Naoúm, NG (1928), 306 (Fig 33) 2) Perry (1963), pp 19–35 3) A.M. Ball, "Solid Propellants", **AMCP 706–175** (1964), pp 71 & 104

Extrusion of Metals, Plastics, Explosives and Propellants. Extrusion is the process of forcing (by using pressure) a material in softened (plastic) condition thru a suitable orifice in order to produce a body of uniform desired cross-section, continuously. In order to render the material plastic, it is either preheated (as in the case of plastics, solventless smokeless propellant and some metals) or softened by adding a plasticizer (as in the case of some plastics and smokeless propellant). Extrusion may be considered as one of the branches of *size adjustment*

Materials which are usually extruded include: some soft metals (lead, tin, copper, brass, aluminum, magnesium and various alloys); rubber, soaps, ceramics, foods, plastics, Dynamites, Explosives (like Amatol or Explosive D), and Smokeless Propellants. Most of the literature on extrusion is found in books on plastics

For extrusion of conventional American Smokeless Propellants, the gelatinized mass of NC, blended with other ingredients, is charged into the so-called *macaroni press*, from which it is extruded thru a series of finely perforated plates. The pressure, supplied by means of a hydraulic ram, is between 2500 and 3800psi. This operation removes any solid foreign matter and aids the colloiding action of the solvent by further mixing and kneading. The material comes from the press in strands having the appearance of macaroni. The strands are allowed to fall into the *final blocking press* where the material is compressed hydraulically at about 3500psi into a solid block. This operation

FIG 5 MACARONI and BLOCKING PRESSES

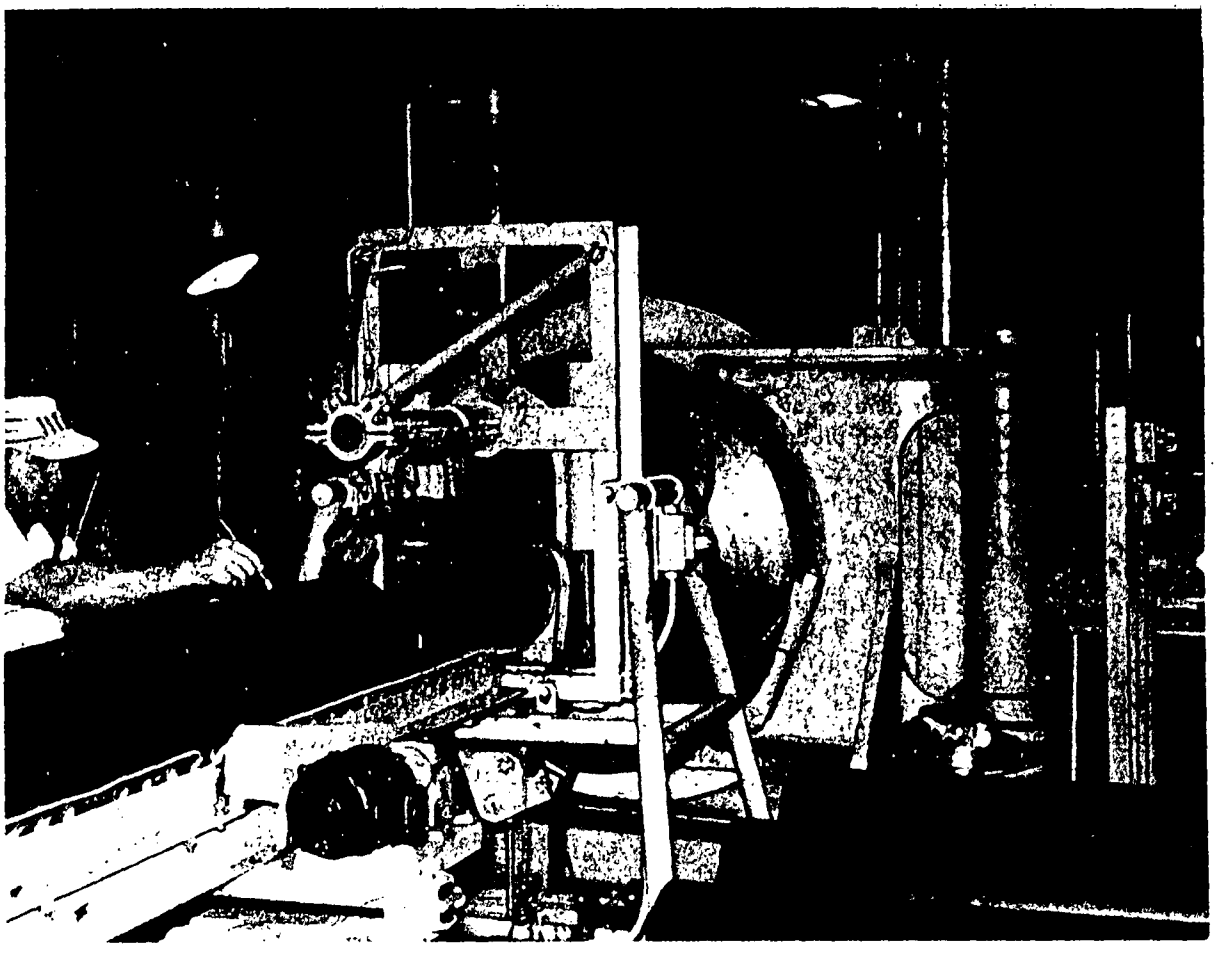


FIG 6 EXTRUSION PRESS

completes the colloiding of the NC, and the material at this point is free from lumps of uncolloided NC and is of uniform appearance

The final block is placed into a *graining press* (finishing press) which may be either horizontal or vertical and from which it is extruded at pressures of from 2500 to 3800psi thru dies fashioned to give the proper cross-section to the grains. The extruded material takes the form of flexible cylindrical cords with one or seven perforations. The press head may be equipped with one or more dies, depending upon the capacity of the press. When small arms propellant is being grained, there may be 36 strands issuing simultaneously from the head. However, with large caliber proplnt, such as for 16-inch gun, it is customary to use but one die in the head. Water is run around the die to cool it during the operation. The proplnt issuing from the press is either led over pulleys directly to a cutter, or allowed to coil up in a basket from which it is fed to the cutter. The latter is equipped with revolving knives which cut the proplnt cord into the desired lengths, as it is fed against the knives by means of feed rolls

Brunswig (Ref 1) & Stettbacher (Ref 2) describe European methods of extrusion of cannon and small arms smokeless proplnts. A booklet issued by CALTEC (Ref 5) gives a brief description of the extrusion of Rocket Propellants

Until WWII, only solvent proplnts were

extruded in the US. The first attempt to extrude Solventless Propellant was made in 1941. For this, trench-mortar proplnt of Picatinny Arsenal, produced in sheets 0.024 inch in thickness by hot rolling process (without the use of solvents) was extruded, using a horizontal press with a barrel 3 inches in diameter and a 0.875-inch die. A pressure-barrel temperature of 170–180°F was used, and a pressure of 3500psi. Better bonded proplnt was obtained with a vertical 3-inch press, at 6000psi and 130°F barrel temperature. From that time, many lots of rocket propellants have been produced by solventless extrusion

Refs (on Extrusion): 1) H. Brunswig, "Das rauchlose Pulver", W. deGruyter, Berlin (1926), pp 85–9 2) A. Stettbacher, "Schiess- und Sprengstoffe", J.A. Barth, Leipzig (1933), pp195–96 3) T.J. Hayes, "Elements of Ordnance", J. Wiley, NY (1938), pp 23–5 4) Anon, "Military Explosives', US War Dept, Technical Manual **TM 9–2900**, Washington, DC (1940), p 13 5) L.A. Burrows et al, USP 2257104 (1941) (Extrusion method to be used for thermoplastic cellulose derivatives) 6) T.L. Davis, "The Chemistry of Powder and Explosives", J. Wiley, NY (1943), pp 302–04 7) California Institute of Technology, "Processing of Rocket Propellants", Pasadena, Calif (1946), pp 1–26 8) J.H. Perry, Edit, "Chemical Engineers' Handbook" McGraw-Hill, NY (1950), pp 1180–83: C.M. Fields & P.A. Crane, "Extrusion" (numerous refs) 9) P. Arribut et al, MP **32**, 279–85 (1950) (Industrial extrusion of propellants under vacuum, without solvents) 10) R.E. Kirk & D.F. Othmer, Edits, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **6** (1964), pp 74–87; Vol **11** (1953), pp 761–68; and Suppl **2** (1960), 709–15 11) H.R. Simonds, H.J. Weith & Wm. Schack, "Extrusion", Reinhold, NY (1952) 12) W.E. Piper, USP 2655694 (1953) & CA **48**, 374 (1954) (Extrusion of fissure-free grains of propellants) 13) A.M. Ball, "Solid Propellants", **AMCP 706–175** (1964), Chapters 8 & 9 14) V. Lindner, "Propellants" in K & O **8** (1965), pp 702 & 710

Exudation in Explosives. (Exsudation in Fr; Ausschwitzung in Ger). Exudation is the act of *sweating* out liquid ingredients from an expl composition thru the pores of the solid components. This phenomenon is usually observed in Dynamites (especially those contg large precentage of "explosive oils", such as NG (Nitroglycerin) and NGc (Nitroglycol) or in impure aromatic nitrohydrocarbons, such as Grade II TNT (Setting Point 80.2°) used for loading shells during WWI

In the case of Straight Dynamites, the liquid is held by capillarity or adsorption by the solids, such as wood pulp, sodium nitrate, etc. These expls are the most liable to exude

In the case of Gelatin Dynamites, the liquid is held mainly thru the formation of a colloid and these do not exude unless they are subjected to high pressures or temperatures. However, if gelatins contain an insufficient amount of NC, or if the NC is of unsuitable character (too high nitrogen content, for instance), some liquid which is not a part of a colloid can be squeezed out by applying moderate pressures or temperatures slightly above room temperature. Some blasting gelatins require so little pressure to deprive them of part of their NG, that the pressure caused by weight of the cartridges in the box is sufficient to cause exudation

Exudation is very undesirable from the point of view of safety because free NG or NGc is much more sensitive than Dynamite. Moreover, the loss of NG or NGc by exudation diminishes the strength of Dynamites, as well as their sensitiveness to detonation

It is the general rule in the US that all Dynamites showing exudation are destroyed at once and that any surface with which the exuding liquid has come in contact is chemically cleansed or destroyed as a dangerous article

Exudation in Shells Loaded with Grade II TNT and Amatol. Shortly after WWI, when large amounts of TNT and Amatol-loaded shells were stored, it was noticed that the TNT and 50/50 Amatol-loaded shells seemed

to be undergoing some sort of deterioriation, evidenced by the exudation of black, oily, tarry material and, in some cases, a gas

Investigation showed that the principal causes of exudation were:

1) The presence of impurities in the TNT
2) The use of alcohol shellac in the booster cavity, and alcohol for cleaning the threads in the nose of the shell
3) In the case of Amatol shells, the introduction of impurities contained in the technical Ammonium Nitrate (AN)

The principal impurities in item (1) were the undesirable isomers of TNT & DNT; their presence lowered the mp (Setting Point) of α-TNT from 80.75° to 80.2° (Specification grade) and could lower it much more. On casting TNT in the shell, the outside of the cast solidified first because of the cooling effect produced by the metal walls of the shell, while the center portion of the cast solidified last, because it was somewhat insulated. As the result of this cooling, the mixture that solidified at the outside contained a large proportion of high melting components, while the portion that solidified in the center core contained the bulk of the impurities because they solidified at a lower temperature. If such shells were stored at elevated temperature(in tropical countries or in the deep South of the United States), the low melting point areas would liquefy and, due to expansion, force their way out thru the threads of the booster cup

All this was purely physical action and no chemical reaction took place and no gas evolved, unless alcohol had been used. When alcohol was used, as in shellac for the booster cavity or for washing the threads, in the nose of the shell, some chemical reaction took place and not only exudate was formed but gas as well. This gas has proved to be Ethyl Nitrite. By laboratory experiments, it was determined that TNT and alcohol do react and give the same gas and tarry material as that found in the shells

As a remedy, it is advisable to use TNT of a higher setting point than 80.2° and to use no alcohol

Regarding the exudation due to impurities introduced by technical AN in TNT mixtures, it should be noted that this problem existed when AN was prepd from ammonia obtained in the destructive distillation of coal. This AN contained many organic impurities (such as pyridine) which have been found to act in a manner similar to alcohol

It was noted that 50/50 Amatol using impure AN, exuded, while 80/20 Amatol (AN 80 & TNT 20%) seldom did. This was because of the comparatively small amount of TNT present and because the AN absorbed most of the exudate. With the introduction of AN prepd from synthetic ammonia (as was done during WWII) and the use of better grade TNT, the exudation of Amatols nearly stopped

In addition to the black tarry exudate, some samples of TNT and Amatol contained the red particles of an expl compound which contained iron. It was shown that this material was the result of reaction of TNT and alcohol in the presence of iron. The iron came from corrosion of the shell by the exudate, in the case of TNT, or by moisture in the AN. Several methods were introduced to prevent rusting of the shell, one of them by coating the inside of the shell with acid-proof paint, another was by controlling the moisture in the AN and still another by pouring on top of the Amatol some TNT as a "booster-surround". The purpose of the latter was to seal the hygroscopic AN from moisture

Exudation is very undesirable because it is a fire hazard, causes corrosion of the shell and also causes cavitation in the charge

It is a fire hazard because, when the exudate collects on the floor of a storage magazine, it might accidently catch fire, especially if it contains red particles of the expl iron salt

Corrosion of the shell case & booster cup is caused because the exudate is often acid in reaction. When exudation is noticed in any shell, the booster should be removed and the cavity cleaned

Cavitation in the filler often resulted from exudation. It could cause trouble in two ways: first, by desensitizing the booster or the surrounding charge and, second, by

lowering the density of the loading, thereby decreasing the efficiency of the shell. Although the exudate contains TNT, it is much less sensitive to detonation, so that the booster is less liable to cause a high order detonation when initiated by a standard fuze, such as the Mark III. This can be remedied, however, by using the so-called "Horse Detonator" (qv)

It was assumed at one time that cavitation was dangerous since, during set-back, a premature might occur because of the crushing of one piece of TNT against another, or against the shell wall, or the pinching of TNT exudate in the booster threads. Investigations, conducted with 75 and 155mm shells which were in a very bad state of deterioration from exudation, showed that, even if these shells were fired at 12% excess pressure in the gun to make the worst possible combination of conditions, no prematures resulted either in the gun or along the trajectory. It was, therefore, concluded that with those two caliber guns, it was safe to use exuding shell. The restriction in use of such shells was therefore removed

Post-WWII investigations conducted at Picatinny Arsenal showed that a dark reddish-brown exudate collected from 105mm shells loaded with TNT consisted principally of a mixture of α-TNT with some β- and γ-TNT and, sometimes, traces of 2,4-DNT and other products of nitration of impurities in toluene. It is also probable that alcohol present in "NRC" sealing compound and/or other extraneous material might favor exudation. The exudate had a much lower stability and brisance than α-TNT but the same sensitivity to friction or impact. In some of the 105mm shells as much as 39.5g of exudate was collected, which amounts to 1.8% of total TNT present (Ref 5)

Refs: 1) Daniel (1902), 292–93 (Exsudation) 2) Marshall **1**(1917), 367 (Exudation) 2a) G.C. Hale, ArmyOrdn **4**, 83–4(1923) (High expls and shell exudation) 3) Ch.E. Munroe & J.E. Tiffany, USBurMines Bull **346** (1931), p 25 (Exudation in Dynamites) 4) Davis (1943), 144 (Exudation of TNT) 5) O E. Sheffield, PATR **1749**(1949) (Exudation of TNT in 105mm shells)

Addnl Refs: A) G.D. Clift & S. Fleischnick, PATR **2125**(1955) (Exudation from Cast Explosives) B) S.D. Stein, PATR **2493**(1958) (Exudation in TNT) C) R.W. Heinemann, PATR **2568**(1958) (Control of Exudation by Absorbents) D) R.W. Heinemann, Feltman Research and Engineering Laboratories, Explosives Development Section, FREL, EDS **No 65**, Picatinny Arsenal, Dover, NJ, May 1959 (Control of Exudation by Absorbents)

Exudation Tests in Dynamites. Several tests are known. They were designed to determine whether Dynamites would remain unchanged in their composition under adverse conditions of storage

The earliest description of exudation tests at our disposal is in the book of Daniel (Ref 1) in which are described the following tests:

a) British Government Test (probably obsolete) consisting of freezing and melting a weighed cartridge, repeated 3 times. No exudation should take place (Ref 1, p 292)

b) British Government Test, described in Daniel as *Liquéfaction (Essai à)* (p 409), was intended to determine exudation or softening of gelatinous expls stored at temperatures in hold of a ship. As such expls do not respond to Centrifuge Test, it is known that if not properly made they show exudation. This may be due to liquefaction of a portion of the gel and not merely to separation of NG from the NC and other components of the dope. A Gelatin Dynamite in this condition is unsafe to use. The test prescribed by HM Inspectors (Ref 1, p 37) was as follows: After stripping the wrapper from the cartridge of expl selected for the test, a section equal in length to its diam is cut transversely so that the ends are flat and the edges sharp. This is then pinned with its axis vertical to a flat wooden or cardboard surface and placed in an oven maintained at temp 85° to 90°F(29.4° to 32.4°C). After exposure of 144 consecutive hours (6 days and nights), the sample is removed and examined. It passes the test if the upper surface

is still flat with the edges still sharp and the diminution of height did not exceed 25%. Same test is described in Marshall (Ref 3) and Barnett (Ref 4)

c) Another test, described by Daniel (p 293), was intended to determine exudation under moderate pressure, which is encountered during loading of Dynamites into cartridges. For this test a brass tube of the same diameter as cartridges was used. The tube was pierced with many tiny perforations and closed at the bottom. Thru its open top end, a cartridge was inserted all the way to the bottom and on top of it was placed a piston of a press which could develop pressures of 4–5kg/sq cm. If no exuded liquid appeared around the perforations, the Dynamite was considered to pass the test

Note: It seems that the test could be made quantitative if the tube were wrapped, prior to the test, with one layer of tared filter paper. Then, if the paper were weighed after the test, exudate could be calculated

A more recent French test called "Exsudation par étuvage" is described by Médard (Ref 6) and in Vol 1 of Encycl, p XI. A similar French test was described by Pepin Lehalleur (Ref 5, p 61) who also described the Pressure Test similar to that described in Daniel and the Centrifuge Test similar to that described in Refs 2 & 3 and in Vol 2 of Encycl, pp C143-R & C144-L

Snelling & Storm (Ref 2, pp 8–11) described the following tests, used at that time (1916) at the USBurMines: a) Forty Degree Test b) Pressure Test and c) Centrifugal Test. Then the Pressure Test was abandoned and in Bulletin 346 of 1931 (Ref 4, pp 25–6), only Centrifuge Test and Forty-Degree Test are described, in addn to the British Test (pp 26–7). Centrifuge Test is described in Vol 2 of Encycl, pp C143-R & C144-L, while Forty-Degree Test is in this Vol. The British Test is described as item b under tests described in Daniel (Ref 1, p 409)

Refs: 1) Daniel (1902), 292–93 & 409 2) W.O. Snelling & C.G. Storm, USBurMines Bulletin 51 (1916), 8–11 3) Marshall 2 (1917), 419–22 3) Barnett (1919), 215 4) Ch.E. Munroe & J.E.Tiffany, USBurMines Bull 346 (1931), 25–7 5) Pepin Lehalleur (1935), 61 6) L. Médard, 33, 328 (1951) *Addnl Refs:* A) E. Burlot, MAF 14, 303 (1935) (Exudation test) B) Explosives Research Laboratory, Bruceton, Pennsylvania, "Interim Report on the Preparation and Testing of Explosives", Division 8, NDRC (1944) (Exudation Test – Percent loss at 150° F in 1 week determined by heating a weighed cast or pressed sample on 10 layers of filter paper placed in a container provided with a cover and heated in a constant temp oven. The loss of weight indicated the extent of exudation) C) L. Médard, MP 33, 501 (1951) (Exudation test)

Exudation Tests for TNT and Amatols. The following test, called Ausschwitzungsprobe, was used in Germany during WWII: A 20-g sample of TNT, melted and cast as cylinder 18mm in diam, was placed with the flat bottom part on a sheet of special Schleicher & Schülle filter paper resting on an aluminum plate. As a reference "standard", a similar pellet of Grade A TNT (setting point 80.6°) was placed on the same sheet but about 100mm away. The ensemble was placed in an oven and left there for 6 hours at 72°. After cooling, the diameter of the circle produced by exudate was measured and, if it were not larger than 35mm, the TNT was considered as Grade A. Any diam betw 35 and 70mm was considered as Grade B (sp 79.5°). In addition to these two grades, the Germans manufactured Grade UK (Umkrystalliziert=recrystallized) with a sp of 80.7° to 80.8° (Refs 1, 2 & 4)

Sheffield (Ref 3) used the following test during his investigation of exudation of TNT in 105mm artillery shells conducted at Picatinny Arsenal after WWII: A portion of TNT was melted and cast in the form of discs about 1 cm thick and 5mm in diam. After removing the rough spots, the disc was weighed and placed on 2–3 layers of hard, previously weighed, filter paper located in a flat aluminum dish (such as used for determination of moisture in NC), provided with a cover. After heating the ensemble at 71°C (160°F)

for 16 hours, the disc was separated from paper while still at 71°, then cooled and reweighed. The same was done with filter papers and, in addition, the diam of exudate spot was measured

A summary of the pertinent results from exudation studies conducted at PicArsn was prepd by Stein (Ref 5). In one series of tests to induce exudation and to obtain exudate samples for analysis and examination of properties, a loaded unfuzed shell was placed in an inverted position over a tared dish and maintained for several hours at 160°F (71°C), until exudation stopped. Then the dish was reweighed and % of exudate calcd

Refs: 1) C.H. Brooks, PB Rept **22930**(1945), 15 2) O.W. Stickland, PB Rept **1820**(1945), 7 3) O.E. Sheffield, PATR **1749**(1949) 4) B.T. Fedoroff et al, PATR **2510**(1958), p Ger 45 5) S.D. Stein, PATR **2493**(1958) 6) B.T. Fedoroff & O.E. Sheffield, PATR **2700**, Vol **1**(1960), p XI, PB No. 171603(I)

Exudation in Cast Explosives in Presence of Crack-Preventing Additives. See under "Anticracking Additives to Cast Explosives" in Vol **1**, pp A461-R to A462-R

Eye Bombs (US Navy). The US Navy has under development or production what it calls its "Eye" series of bombs. The Snakeye is a 250 to 500 low drag bomb. (See Vol **4**, this Encycl, pp D940-R & D941-L). The Rockeye is a new cluster bomb which can contain either anti-personnel or antitank bomblets. The Fireye is a new incendiary bomb and the Sadeye is another cluster bomb designed to be air-launched. It contains a large number of bomblets. The Weteye is a new chemical bomb of the non-lethal variety designed to replace the Navy MK 94

Ref: R.L. Johnston, "Air Armament", Ordnance **61**, 121 (1966)

Eyring Absolute Reaction Rate Theory. See "Absolute Rate Theory" in Vol **1** of Encycl, p A4-R and in Cook (1958), p 134

Eyring Detonation Theory. See "Detonation, Curved Front Theory of Eyring et al" in Vol **4** of Encycl, pp D242-R to D244-L

F

"f". Force or energy of expls, which is also called "specific pressure" (In French, force spécifique). "f" is the pressure (in atm) per unit area (cm^2) that would be obtained by the expln of unit weight (1 g) in unit volume (1 cc), if the ordinary gas laws were applicable at that density

Following is the formula, according to Abel:

$$f = pv = \frac{V_o Q}{C} \times \frac{p_o}{273} + \frac{p_o V_o T_1}{273} = p_o V_o \frac{(Q/C + T_1)}{273} \text{ atm}$$

where:

p = pressure of expln in atmospheres
v = volume of vessel in which expln takes place
V_o = specific volume of the gaseous products (volume of gases developed on expln) of unit wt of expl (calculated at 0° and standard pressure p_o), expressed in cc/g
Q = quantity of heat set free by the expln of unit weight
C = sum of mean specific heats of the products of expln, expressed cal/g
T_1 = absolute temperature of the expl before it is fired, which may be considered equal to 273°

In order to obtain f expressed in kg/cm^2, the above value has to be multiplied by 1.0333

On substituting in the above formula, the value Q/C by t = maximum temperature of expln, and T_1 by 273°

$$f = p_o V_o \times \frac{t + 273}{273} \text{ atm}$$

According to the kinetic theory of gases, the kinetic energy of the expl gases is equal to 1.5f

Berthelot used the expression $V_o Q/C$, which he called *"characteristic product"* (Refs 3 & 4) to measure the power of the expl, but due to the difficulties of determination of C, the product $V_o Q$ has often been used instead. However, both of these expressions are unsuitable for expressing the power of expls, as can be seen from some comparative values given on p B105-R of Ref 4. However, this does not prove conclusively that the above expressions are without practical significance

Refs: 1) Marshall **2**(1917), 467–8 2) Vennin, Burlot & Lécorché (1932), 53 (Force théorique) 3) Pepin Lehalleur (1935), 40–50 4) Encycl, Vol **2**(1962), pp B105 & B106 (Berthelot's Characteristic Product) and p B265-R, under BRISANCE OR SHATTERING EFFECT

"F" (Dynamit). See Dynamit "F" (Swiss) in Vol **3** of Encycl, p C443-L, under "Swiss Commercial Explosives of Non-permissible Type"

F (Propellants). British Cordite type proplnts of various compositions:

a) F/478/138/K: NC(wood) 56.0, NG 43.0, Centr 1.0, K cryolite 2.0, and C black 0.1 (added). FI (qv) 11 (PA) considered low: comp to LA or NG although this proplnt is not considered dangerous to handle

b) F/488/343: NC 53.0, NG 43.5, Centr 1.5, DBuPh 2.0, C black 0.1, and wax 0.075 (added). FI:27. Both proplnts burned w/o deton

c) F/547/18: NC 46.0, NG 23.0, acetylcellulose 12.0, triacetin 18.0, K cryolite 1.0, chalk 0.2, and wax 0.075 (added). This proplnt failed to ignite on impact

Ref: P.W.J. Moore & R. Pape, ERDE Tech Mem No **14/M/54**(U) (1954)

"F" (Propellants). Additional List of British post-WWII propellants:

F428/180. A Brit proplnt with Adiabatic Flame Temperature (AFT) 1950°K: NC 65, NG 15.4, Carbamite (EtCentralite) 2 & DBuPh (Dibutylphthalate) 17.6%

F487/46. Ditto with AFT 1950°K: NC 20, Picrite (Nitroguanidine) (NGu) 60, DEGDN 11.16, EtCentr 2.64 & DBuPh 6.2%

F487/68. Ditto with AFT 1950°K: NC 70, DEGDN 14.3, EtCentr 2 & DBuPh 13.7%

F488/312. Ditto with AFT 2800°K: NC 56.5, NG 33, EtCentr 3.5 & DBuPh 7%

E551/66. Ditto with AFT 2800°K: NC 21, NGu 55, NG 21 & EtCentr 3%

Ref: H.H. Abram, T. Williams & K.F. Allen, Armament Research Establishment Metallurgy Report No **8/54**, Woolwich, SE 18 (1954), Table 1 (1954)

F1. An Austrailian submachine gun characterized by a keen balance, facility of operation and lack of recoil. It is blowback-operated and fires the 9-mm Parabellum cartridge at a cyclic rate of about 650 shots per minute. The 8½-inch barrel delivers a muzzle velocity of about 1250 feet per second, using ammunition compatible with the Browning High Power Australian Service Pistol

The F1 is manufd at Lithgow Arsenal, Lithgow, New South Wales and is issued to AMF (Australian Military Forces) to complement the semiautomatic SLR (Self-Loading Rifle)

Ref: T.L. Golden, Ordn **57**, 228–30 (Nov-Dec 1972)

F33B. Gasless incendiary mixture: Zr (200 mesh) 41.0, Fe_2O_3 (pigment grade, red) 49.0 & "Superfloss" 10%

Note: Superfloss is a trade mark of Johns-Manville Products Corp, NY for a grade of "Celite" (diatomaceous earth)

Ref: Tech Command, Army Chem Center, Maryland, "Development of an Incendiary Pellet", **TCR–59** (May 1950)

FA-878. Frankford Arsenal primer composition consisting of Ba nitrate 20, Pb dioxide 20, granulated Zr 32.5, powdered Zr 7.5 & PETN (grade B) 20%. This mixt is used in small arms cartridges

Ref: Frankford Arsenal, "Primer, Pyrotechnics and Incendiary Compositions for Small Arms", Rev 2 (1954), p 43

Fabric Cordeau. A detonating fuse made from tubes or woven fabric filled with NC, TNT, PETN, etc. See Cord, Detonating in Vol **3**, p C529-R and under Detonating Cords in Vol **3** p D103-R

"Factors Affecting Detonation Velocities of Desensitized Nitroglycerin in Simulated Underground Fractures". Title of paper by J.S. Miller, W.D. Howell, J.L. Eakin & E.R. Inman, issued as RI (Report of Investigation) **7277**, July 1969 by the USBurMines, 4800 Forbes Ave, Pittsburgh, Pa, 15213. This report is reviewed in Expls&Pyrots **2**(10), 1969, under the title "Liquid Explosives Tested for Rock Fracturing"

Factors Influencing Velocity and Other Properties of Explosives. See Vol **4**, p D347-L, under "Detonation, Factors Influencing Velocity and Other Properties of Explosives in"

"Factors in Selecting and Applying Commercial Explosives and Blasting Agents". Title of USBurMines Information Circular **IC 8405**(1968), compiled by R.A. Dick (30 pp). Obtainable from Superintendent of Documents, Govt Printing Office, Washington, DC, 20402 (22 refs). Reviewed in Expls&Pyrots **2**(6), June 1969

Fading or Fadeout of Detonation. See Vol **4**, p D223-R, under Detonation: Attenuation, Break, Cessation, Cutoff, Dying-out, Extinction, Fadeout and Failure

Fading of Detonation in Cones of Explosive. A deton wave once formed in an expl has a tendency to die out or "fade" if the diam of the expl is below a certain min (called *boundary diam*). It may be detd by using a cast cone detonated from the base. The diam of the cone at which the wave is extinguished is a measure of the fading properties of the expl. The observation may be made photographically by means of a rotating mirror camera. The value so obtd tends to be less than the value from cylindrical columns of expl. See Vol **4**, p D223

Ref: D.W. Woodheath & R. Wilson, Nature **167**, 565 (1951)

Fading of Detonation in Solid Explosives was discussed by O.A. Gurton in ProcRoySoc **204A**, 31–2 (1950) & CA **45**, 10585 (1951)

FAE and FAX (Fuel-Air-Explosives). The acronym of the US Fuel-Air Explosives Program which has been changed from FAX to FAE to prevent confusion with Fighter-Aircraft Experimental (FAX) Program. FAE represents the entire fuel-air explosives program, including current projects

Some basic work on the formation of spherical detonation waves from explosing gas mixts confined in ballons was reported by Freiwald & Uhde (Ref 2). Magram in the US earlier studied the effects of expl gases (Ref 1). Many of the first feasibility studies of FAE and FAE weaponization developments were carried out at the US Naval Ordnance Test Station, China Lake, Calif (Refs 3 & 4)

Expl vapor clouds were once considered a way-out technique. A cloud of volatile fuel, mixed with air, is discharged and then detonated on a target, with the same violent expln that characterized grain-silo dust explns or blast. when a propane tank truck blows up. This kind of expln can level a city block. Very significantly for military purposes, it can reach around corners and into structures & shelters, which ordinary HE, fragmentation bombs and shells cannot do (Ref 12)

Fuel in gaseous, liquid or solid phase can be made to detonate in air (Refs 5, 6 & 7). These detonations produce much lower maximum pressures (order of 20 atmospheres) than detonation of high explosives, but can produce larger static and dynamic impulses (defined by

$\int_o^t P\,dt$ and $\int_o^t \tfrac{1}{2}\rho U^2\,dt$

where: P = pressure
t = time
ρ = density
U = velocity

at a fixed position) per weight of fuel and can cause blast effects over larger areas. Essentially this is a consequence of the high energy of explosion per unit weight of fuel (when reacted with atmospheric oxygen) compared to high explosives

More recent development has centered on detonation of liquid fuel drops dispersed in air, although fuel films will also support detonation (Ref 9). In the fuel drop-air explosive, it is necessary that relatively large liquid fuel drops be broken up into a micromist of fuel spray. The fuel spray can then combust rapidly enough to drive the detonation shock. Drop breakup is accomplished by the high speed gas flow associated with the detonation shock. Since breakup is a relatively slow process, the reaction zone of these detonations is large. Losses from this reaction zone can cause the detonation velocity to be less than the theoretical Chapman-Jouguet velocity, with the velocity proportionally lower for larger drop diameters (Ref 8). Both drop size and drop spatial distribution (that is local fuel-air ratio) have important effects on these detonations

Monopropellant drops have been shown to be capable of detonating in a manner similar to liquid hydrocarbon fuels (Ref 11). With monopropellant explosives, energy release per unit volume of explosive is not limited by atmospheric oxygen; however, energy release per unit weight is smaller than for fuels using atmospheric oxygen

FAE have joined chemical & nuclear expls for military use. Vaporized fuel from air-dropped or preplaced canisters, exploded on a target produces highly destructive blast. In Vietnam, FAE cleared out mine fields & booby traps, and opened up helicopter landing fields (Ref 15)

Continued development of high-speed FAE weapons systems now depends on a joint service proposal. The joint service plan identifies funding & specific areas of development. The first bombs developed by the US Navy, CBU-55, were used in Vietnam, and tests to perfect an advanced version of the weapons system have continued. The CBU-55B air-to-surface free-fall cluster bombs were developed for helicopter and slow-speed fixed-wing aircraft delivery to clear helicopter landing zones of mines & booby traps. This FAE bomb was designed to disperse a mixt of vaporized fuel (ethylene oxide, propylene oxide, methylacetylene/propadiene/propane and others) in a cloud 50 ft in diam and approx 8 ft thick. It has three 100-lb individual canisters 13.6 inches in diam by 21 inches long, each containing ca 72 lbs of fuel. The canisters separate from the dispenser after

release from an aircraft, and each is individually retarded by a drogue chute as it nears the target (Ref 13)

The Air Force at its Armament Development & Test Center, Eglin AFB, Florida and the Navy at the Naval Weapons Center, China Lake, Calif have been working separately to perfect a second-generation FAE Weapons system as a bomb for high-performance jet aircraft delivery. The Army, meanwhile, has been involved in developing its own FAE system centered on mine clearance. To assess the terminal effects of the Navy FAE 2 weapon, this second-generation system was placed on a barge and floated near the decommissioned destroyer escort, USS McNutly, anchored off San Clemente Island, Calif in 90 ft of water. The expln caused sufficient damage to sink the ship. Defense officials believe that only the surface has been scratched in developing uses for FAE weapons. Fuel air expls open a potential for increasing blast effects while reducing wt. Guidance seekers can be added for a high-speed version, the warhead size can be increased, the cloud detonation can be incorporated in missile systems, standoff capability & the modular weapons concept all apply to FAE (Ref 13)

Refs: 1) S.J. Magram, "Explosive Gases", ChemCorpsTechCommand, Army Chemical Center, Maryland **TCR-1**, Proj 4-09-04-03 (July 1948) 2) H. Freiwald & H. Uhde, CR **236**, 1741(1953) & **241**, 736(1955) 3) W.A. Gey & M.A. Nygaard, "Feasibility Study of FAX Explosives", NAVWEPS Rept **8065** (Jan 1963) (Conf) 4) R.G. Hippensteel, "Measurement of Blast from a Five-Pound Experimental FAX Charge", Ballistic Res Labs, BRL Memo Rept **1531** (Jan 1964)(Conf) 5) K.W. Ragland, E.K. Dabora & J.A. Nicholls, "Observed Structure of Spray Detonation", Physics of Fluids, Vol **11**, No 11, 2377(1968) 6) W.A. Strauss, "Investigation of the Detonation of Powdered Aluminum-Oxygen Mixtures" AIAA Joumal **6**, 1753(1968) 7) J.H. Lee, R.I. Soloukhin & A.K. Oppenheim, "Current Views on Gaseous Detonations", Astronautica-Acta **14**, 565(1969) 8) E.K. Dabora, K.W. Ragland & J.A. Nicholls, "Drop-Size Effects in Spray Detonation", Twelfth Symposium on Combustion (1969), pp 19-26 9) J.R. Bowen et al, "Heterogeneous Detonation Supported by Fuel Fogs on Films", Thirteenth Symposium on Combustion, The Combustion Institute (1970), pp 1131-39 10) C.W. Kauffman, J.A. Nicholls & K.A. Olzmann, "The Interaction of an Incident Shock Wave with Liquid Fuel Drops", AIAA 9th Aerospace Sciences Meeting, NY (Paper No 71-206) (Jan 25-27, 1971) 11) P.L. Lu & N. Slagg, "Chemical Aspects in the Shock Initiation of Fuel Droplets", Third International Colloquium on Gasdynamics of Explosions and Reactive Systems, AstronauticaActa **17**, 693-702(1972) 12) Staff, The Detroit News, Sect F, p 2F (12 Oct 1972) 13) C.A. Robinson Jr, "Special Report: Fuel Air Explosives", Aviation Week & Space Technology, pp 42-46 (19 Feb 1973) 14) L.H. Josephson, "Mechanisms and Utility of Fuel-Air Explosives", Naval Weapons Center, China Lake, Calif **TP 5444** (Feb 1973) (Not used as a source of info) 15) Staff, AOA Newsletter, "The Common Defense", p 2 (16 April 1973)

Fahneljelm proposed in 1876 a Dynamite known as **Nysébastine** (EnglP 4075). It consisted of NG 45-45, porous charcoal 15-35, nitrate or chlorate of K or Na 5-25 and Na carbonate max 5%

Ref: Daniel, Dict (1902), 294 & 586

Fahrenheit, G.D. (1686-1736). A German physicist who devised meteorological instruments, hydrometers and thermometers.

His thermometer scale is based at the lowest temperature which he could obtain by freezing mixtures. He marked the freezing point of water as 32°F and its boiling point as 212°F. Hence, 1.8°F = 1°C and absolute zero -273°C = -459.4°F

In order to change °F to °C subtract 32 from the °F, multiply by 5 and divide by 9

In order to change °C to °F, multiply the °C by 9, divide by 5, and add 32

Ref: Hackh's (1944), 331-R & 847-R

Fahrm's Explosive (Fr), patented in 1898 & 1899, contained AN 86, NG 5, rosin 5 and K chlorate 4%
Ref: Daniel (1902), 294

Failure Analysis. A rigorous scientific investigation, Failure analysis is an examination of failed parts, operating conditions, factors affecting performance, problem history, and other pertinent factors. Qualified experts in several disciplines can then recommend definite corrective action. One common reason for failure is material properties. Chemical analysis, mechanical testing, or structural studies will usually confirm or eliminate this failure type. Another reason for failure is operation of the part in an environment not contemplated by the designer. In analyzing a material involved in a failure, optical and electron microscopy can be used along with X-ray diffraction analysis, thermal analysis, gas chromatography, etc. If actual mechanical failure has taken place, then fractography can be used to detect overloading, environmental incompatibility, or fatigue
Refs: 1) Laboratories Highlights, Vol **2**, No **1**, May 1972, The Franklin Institute Research Laboratories 2) Expls&Pyrots **5**(8)(1972)

Failure Density. The minimum density below which detonation will fail. See Critical Density, Vol **4**, pp D190 to D193. Also under Detonation Velocity-Charge Density Relationship, Vol **4**, pp D643 to D646 and pp D646 to D652

Failure in Detonation. See Fading or Fadeout of Detonation in Vol **4**, p D223-R

Failure in Detonation of Coal Mining Explosives. See Vol **4**, p D347-R, under "Detonation, Failure of Coal Mining Explosives in a Bore Hole"

Failure Diameter. The minimum diameter below which propagation of detonation does not take place. Also known as critical diameter or limiting diameter. See Vol **4**, p D660-L. Also under Detonation Velocity-Charge Diameter Relationship, Vol **4**, p D641-R and Vol **3**, p C560-R and C561-L. For information on critical diameters of a) Liquid explosives, See Vol **4**, p D197-L and D653-L b) Powdery explosives, See Vol **4**, D653-L c) HEs, See Vol **4**, pp D560–D562

Failure Length of Propagation of Detonation. See Vol **4**, pp D199-L to D201-L under Critical Length of Propagation of Detonation

Fajans, Kasimir (1887–). A Ger physicist noted for his work on radioactive substances and the discovery of radioactive element Bv (brevium), also known as uranium X_2. It has a half-life of only 1.65 mins. He is also known for *Fajans-Soddy Law* which states that when an α-particle is expelled from a radioactive substance the product is two places lower in the periodic table. A β-ray change, or expulsion of an electron, produces a rise of one place
Refs: 1) Hackh's (1944), pp 330-R (Fajans) and 142-R (Brevium) 2) Anon, "The International Who's Who" (1973–74)

Falcon and Falconet
Falcon was a *Culverin* type artillery piece of about 16th century which, accdg to Dupuy & Dupuy (Ref), weighed 800 lbs, had bore of 2.5 inches, length 6.0 ft and fired projectiles weighing 3.0 lbs. Its point blank or effective range was 400 yds and maximum range 2500 yds
Falconet was its smaller version, weighing 500 lbs, having bore of 2.0 inches, length 3.7 ft and weight of projectile 1.0 lb. Effective range was 280 yds and maximum range 1500 yds
Note: Culverins are briefly described in Vol **3** of Encycl, p C573-L
Ref: R.E. Dupuy & T.N. Dupuy, "The Encyclopedia of Military History", Harper & Row, NY & Evanston (1970), p 453 (Table)

Falcon. A USAF air-to-air missile produced by the Hughes Aircraft Co, guided either by its own radar or by infra-red homing, propelled at Mach 3 by a Thiokol Co solid fuel motor. Initially, it had a 5 mile range, 50–70000 foot ceiling, and carried a high expl warhead. A larger model was later developed with longer range and nuclear warhead
Refs: 1) S. Ulanoff, "Illustrated Guide to US Missiles and Rockets", Doubleday, Garden City, NY (1959), 79 2) F.I. Ordway III & R.C. Wakeford, "Internation Missile and Spacecraft Guide", McGraw-Hill, NY (1960), 32–3

Falkenstein, K. von patented in 1892 the smokeless proplnt called *Cibalite.* See Vol **3** of Encycl, p C322-R

Fallenstein & Petry patented in 1884 in England a plastic expl, called *Kinetite.* It consisted of Nitrobenzene 16.00 to 21.00, gelatinized with small quantity (0.75–1.00) of soluble NC, mixed with finely pulverized K chlorate 75.00–82.50 and precipitated Sb sulfide 1.00–3.00%. It was claimed that Sb sulfide served as a regulator of expln
Ref: Daniel (1902), 295 & 392

Fallex. (Abbr for Fall Exercises) A big training exercise without troop movements, carried out each autumn by NATO. Its object is to check on the efficiency and reliability of the communications system of the collective defense organization
Ref: P.G. Thillaud, "Révue Militaire Générale", Berger-Kevrault, Paris, June 1963, pp 7–19

Fallhammerprobe or Fallhammerprüfung (Ger for Falling Hammer Test). See IMPACT SENSITIVITY OR SHOCK SENSITIVITY TEST in Vol **1**, p XVII and FI (Figure of Insensitiveness Test) described on p XII

Additional Refs are listed in Vol **4**, p D304-R

Falling Ball Test, as Conducted at Atlas Chemical Industries, Inc, Valley Forge, Pa, employed a ball weighing 80g and reported results in inches
The following results are listed: Barium Styphnate 12, LMNR (Lead Mononitroresorcinate)16 and KDNBF (Potassium Dinitrobenzofuroxan) 14
Refs: 1) Atlas/Aerospace, **No 8,** Dec 1969. Atlas Chemical Ind, Inc, Valley Forge, Pa 19481 2) Expls&Pyrots **3**(3), 1970

Falling Ball Test as conducted at Kankakee Ordnance Works, Joliet, Ill employed a 8.33g ball for testing impact sensitivity of LA. See under Ball Drop Test in Vol **1**, p A573-L

Falling Weight Test. Same as Drop Weight Test. See Impact Sensitivity or Shock Sensitivity Test in Vol **1**, p XVII. See also list of Refs on Impact in Vol **4**, p D391-R

Fallout. See under Atomic Energy in Vol **1**, p A502-L

False Ogive. See *Windshield,* listed under Ballistic Cap in Vol **1**, p B5-R

Fanno Line and Rayleigh-Mikhel'son Line. See Vol **4**, p D348-L under Detonation, Fanno Line

Fans and Blowers. *Fans* are devices for transferring vapors, air or other gases for the purpose of ventilation, supplying draft to boilers and furnaces, moving large volumes of gas thru ducts, supplying air for drying, conveying materials suspended in a gas stream, removing fumes, etc. Fans may be classified as low pressure compressors (be-

low 0.5psi pressure head). The most common types of fans are centrifugal and axial-flow

Blowers (such as turboblowers and rotary blowers) are compressors for handling large volumes of gases at pressures from 0.5psi to about 40psi. (See also "Compressors" in Vol 3, p C494)

Ref: J.H. Perry, Edit, "Chemical Engineers' Handbook", McGraw-Hill, NY (1950), 1447–50 and in new edition (1963), pp 6-16 to 6-20

Farad. A unit of electrical capacity; the capacity of a condenser charged to a potential of one volt by one coulomb of electricity. It is 10^{-9} CGS (centimeter-gram-second) unit

Ref: Hackh's (1944), 331-R & 332-L; 50-L (ampere), 229-R (coulomb), 590-R (ohm) and 899-R (volt)

Faraday (F). The quantity of electricity which liberates one gram equivalent of a metal in electrolysis. 1F = 96489 coulombs = 6.06×10^{23} electrons per mole

Ref: Hackh's (1944), 332-L

Faraday, Michael (1791–1867). A Brit chemist and physicist noted for liquefaction of gases, the effect of electric current on the magnetic needle, electrolysis, and as discoverer of benzene. He also introduced the law stating that the wt of an ion deposited electrolytically is proportional to the strength of the current passing thru the soln

The *washing bottle*, currently used in labs was his invention and so was *ammonia tube*, a V-shaped tube of strong glass which, inverted, is used for distn, purification and crystallization of liquids under pressure

Ref: Hackh's (1944), 332-L

Farmer's Dynamite. An inexpensive Brit 40% strength expl (See Strength of Explosives in Vol 1 of Encycl, p XXIV) employed for breaking up sub-soil, making holes for trees, poles or other agricultural purposes. It contains NG $\leqslant$39.5 (gelatinized with NC $\geqslant$0.75) absorbed by dry wood meal $\geqslant$16.5, $NaNO_3$ and $MgCO_3$ 1%. It differs from ordinary American 40% straight dynamite in being less subject to exudation because the NG is gelatinized and a larger percentage of wood meal is used

Ref: Marshall 1 (1917), 370

Farmer's Test for Stability. See Vacuum Stability Test in Vol 1 of Encycl, p XXVI

Fasan (Pheasant) **Powder.** One of the varieties of older German shotgun propellants. It consisted of greyish-yel round grains and the charge was 2.65g. Specially designed for shooting pheasants

Ref: Marshall 1(1917), 330–31 (Included is microgram)

FASP. In Rocket Technology it stands for "Final Average Sustained Pressure", and refers to the average pressure near the end of a test run before the pressure has begun to fall off markedly

Ref: F. Bellinger et al, IEC **38**, 166 (1946)

Fast Burning Gasless Mixture, which contains: CuO 80 & Al (grained) 20%, is suitable as an incendiary. It develops high pressure on fast burning by sublimation of copper

Ref: W.A. Show, "Development of an Incendiary Pellet", **TCR–59**, Technical Command, Army Chemical Center, Maryland (5 May, 1950)

Fast Neutron Activation Facility at Picatinny Arsenal. A new fast neutron activation facility installed at PicArsn consists of: 1) A Kaman Nuclear Model A711 sealed-tube accelerator for the generation of 14 MEV neutrons by the deuterium-tritium reaction 2) A dual-axis rotating head for the uniform irradiation of samples 3) A sample transfer for the automatic transfer of explosive samples to the accelerator target and back to count station at speeds of 50 ft/sec 4) Two

matched 3-inch x 3-inch sodium iodide scintillation crystals in a large lead shield and 5) Various associated nuclear counting equipment. It is used primarily for rapid, non-destructive quantitative determination of macro quantities of elements (nitrogen, oxygen, lead) in explosives. Trace concentrations of elements can be determined, and the neutron generator can be used as a neutron source for radiation studies

Refs: 1) O.E. Sheffield, ExplsLabNews **3** (Nov 1968), PicArsn, Dover, NJ, 07801 2) Expls&Pyrots **2**(1)(1969)

Fast Photography. See CAMERAS, HIGH-SPEED PHOTOGRAPHIC in Vol **2**, pp C13-L to C19-R

Fast Reactions in Solids. They can be considered from an exptl standpoint to be complete in less than one msec. Low intensity initiation (heat, shock, light, ionizing radiation, "spontaneous") can give rise to a series of stages of reaction, each of which can be terminal or skipped, which lend themselves to study with the proper instrumentation and technique: initiation of decompn in a definite region, change from decompn region to burning region, acceleration of burning and sharp transition to low-vel deton, propagation of low-vel deton, change to high-vel deton, propagation of high-vel deton. Useful tools in the slower stages are electron microscopy and electron diffraction, while high speed photography and electronic methods can be used in the later stages

Ref: F.P. Bowden & A.D. Yoffe, "Fast Reactions in Solids", Butterworths, London (1958), 164pp

Fats, Fatty Oils, Tallows, Butters, Waxes and Fatty Acids are general terms for greasy, solid or liquid substances occuring in animals and vegetables

In composition, they are glycerin esters of one or several fatty acids, such as lauric, oleic, palmitic, stearic, etc. Fats are neutral substances, insoluble in water but soluble in ether. *Fatty oils* are fats that are liquid at ordinary temperatures, while *tallows* and *butters* are solid or semisolid at ordinary temperatures. There is no rigid distinction among these terms; for example, "cocoanut oil" is often called "cocoanut butter". Fatty oils are sometimes called "fixed oils" to distinguish them from volatile, ethereal or essential oils. There are also mineral, or petroleum, oils which differ from fatty oils in composition but possess some common properties. For instance, both fatty oils (such as castor oil) and mineral oils possess lubricating properties

Waxes are solid or semisolid plastic substances consisting of mixtures of esters, fatty acids, hi gh molecular-weight alcohols and even hydrocarbons. Waxes differ from fats in that they are the esters of monohydric, high molecular weight alcohols, whereas fats are the esters of trihydric, low molecular weight alcohols, such as glycerin. Examples of waxes: beeswax, carnauba, spermaceti etc. There are also substances obtained from mineral sources which resemble waxes in appearance and in some properties, such as paraffin, ceresin, montan wax, etc

Fatty acids are aliphatic monocarboxylic acids, many of which occur as esters of glycerin (glycerides) in natural fats and oils. For example, acetic, butyric, propionic, lauric, myristic, palmitic, margaric, stearic, oleic, linolenic, ricinoleic, etc

Some fats, oils and waxes have been used as ingredients in explosive compositions, such as plastic explosives. They can also be nitrated to give nitro- or nitrate compounds

Refs: 1) T.P. Hiditch, "The Chemical Constitution of Natural Fats", Chapman & Hall, London (1947) 2) H. Bennett, "Industrial Waxes", Vol **1**: Natural and Synthetic Waxes"; Vol **2**: "Compounded Waxes and Technology", ChemPubgCo, NY (1963) 3) L.V. Cocks & C. von Rede, "Laboratory Handbook for Oil and Fat Analysis", Academic Press, NY (1966) 4) R.T. Holman, Ed, "Progress in the Chemicstry of Fats and Other Lipids", Pergamon Press, London (Continuing series)

Refs for Nitration:

1) E. Montignie, BullFr [5], **1**, 291–2(1934)

(Cholesterol) 2) S.S. Nametkin et al, Trudy-InstNefti, AkadNauk **2**, 3–9 (1952) & CA **49**, 828 (1955) (C_{18} & $C_{36}\alpha$-hydrocarbons) 3) W. Grimme et al, GerP 847000 (1952) & CA **52**, 11897 (1958) (Aliphatic nitromonocarboxylic acids) 4) Armour & Co, Fr addn 78697 to FrP 1218496 (1960) & CA **58**, 12424 (1963) (Adipic acid) 5) A.L. Dol'berg & A.S. Grishaeva, Khim & Tekhnol Topliv i Masel **9**(9), 33–37 (1964) & CA **61**, 14440 (1964) (Diesel oil) 6) E. Liiv, ZhPrikl-Khim **35**(12), 2770–78 (1962) & CA **61**, 15891 (1964) (Shale oil) 7) F. Asinger, Magy-KemLapja **21**(5), 227–36 (1966) (Hung) & CA **65**, 6969 (1966) (Review with 40 refs) (Substitution reactions of paraffin hydrocarbons) 8) L.F. Albright, ChemEngrg **73**(12), 149–56 (1966) & CA **65**, 6970 (1966) (Nitration of paraffins) (Review with 45 refs) 9) T. Yoshida & K. Namba, KogyoKayakuKyokaishi **27**(3), 139–52 (1966) & CA **65**, 16847 (1966) (Nitration of aliphatic hydrocarbons) (Review with 19 refs) 10) V.Y. Shtern, KhimKinetTsepnyeReakts, InstKhimFizAkadNauk **1966**, 286–322 & CA **66**, 54671 (1967) (Review with 33 refs) (Vapor phase nitration of alkanes by nitrogen dioxide) 11) S.E. Klein, Y.N. Shekhter, "Nitrovannye Masla" (Nitration of Oil), Khimiya, Moscow (1967) 12) T. Yoshida & K. Namba, SekiyuGakkaiShi **10**(8), 562–67 (1967) & CA **68**, 104264 (1968) (Nitration of hydrocarbons) (Review with 61 refs) 13) R.D. Smetana & H. Chafetz, USP 3417127 (1968) & CA **70**, 57139 (1969) (Nitration of hydrocarbons with nitric acid–trifluoroacetic anhydride mixts)

Faure, Mackie & Trench patented in 1873 in England an expl based on NC to which were added rosin, lacquer, ozokerite, collodion, glycerin, and charcoal or soot. The same inventors patented in 1876 expls in which NC was substituted by nitrated sainfoin, esparto grass, agave, hemp, flax, straw, hay, Amer aloe and yucca
Ref: Daniel (1902), 295 & 773 (Trench, Faure et Mackie)

Faustpatrone (Ger for Fist Cartridge), also known as "German Recoilless Grenade Discharger". A shaped charge antitank rocket fired from a tubular discharger, used during WWII against tank. The smaller model, *Faustpatrone 1*, was later called *Panzerfaust 30, Klein*, while the larger model, *Faustpatrone 2*, was called *Panzerfaust 60* and it weighed only 13¼ lbs. Warheads of each model contd HE shaped charges, called in Ger Hohlladungen (Hollow Charges). These weapons could be fired by one man, similarly to *Bazooka*, developed in 1942 in USA (See Vol **2** of Encycl, p B26). More detailed description of Faustpatrone is given in Ref (See Fig 1)
Ref: Fedoroff et al, PATR **2510** (1958), p Ger 46 (Includes 7 refs)

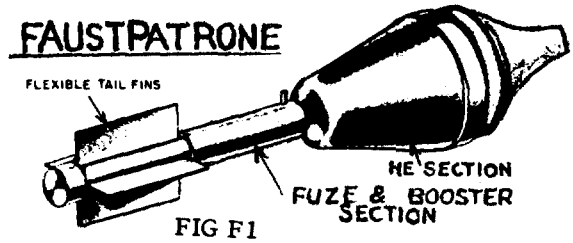


FIG F1

Faversham Mixture, called by Daniel *Fulmicoton Nitraté de Faversham*, consisted of NC 51.6 & Ba nitrate 48.4%. This compn was practically the same as *Tonite No 1*, patented in 1874 in England by Trench, Faure & Mackie, which contd NC 51 & Ba nitrate 49%. There was also *Tonite No 2*, which contd as the 3rd ingredient charcoal and *Tonite No 3* (patented by Trench in 1889), which contd NC 14.55 to 19.00, m-DNB 13.20 to 13.00 & Ba nitrate 72.25 to 68.00%. Some of these mixts were manufd in England, Belgium and USA. The compn of Belgian mixt was: NC 50, Ba nitrate 40 & saltpeter 10%. These mixts were suitable for underwater explns and some of them were used for loading torpedoes
Ref: Daniel (1902), 769–70 (Tonite or Fulmicoton Nitraté de Faversham)

Faversham Powders. Several of the Brit Favier type permissible expls, mfd by the Cotton Powder Co Ltd in Kent, England, initially under the formula: AN 85, DNB 11, NH_4Cl 1.5, NaCl 2.5%. This was later

altered to *No 1:* AN 84–86, TNT 10–12, NH_4Cl 1–2, NaCl 1–3, water 0–2.5%; and *No 2:* AN 87–93, DNT 9–11, water 0–1%. Another formula also called *No 2* was AN 47.5, KNO_3 24, TNT 10, NH_4Cl 18.5%; this one has a limit charge of 24 oz and power (swing of Bal Pend) is 2.61 inches, compared with 2.7–3.1 inches for 8 oz of a 40% American Straight Dynamite

Accdg to Ramsey & Weston (Ref 4), Faversham Powder was practically flameless, a property which was achieved by impregnating the components with the so-called Trench's Fire Extinguishing Compound which consisted of sawdust treated with solns of Amm sulfate or sulfamide. This gave for Faversham Powder: AN 85, DNB 10 & Trench's Compound 5%

Refs: 1) Daniel (1902), 295 2) Thorpe 2 (1912), 416 3) Marshall 1 (1917), 390 4) Ramsey & Weston (1917), 181 5) Colver (1918), 142, 169 & 249

FAVIER EXPLOSIVES (Belg, Fr & Brit). Accdg to Daniel (Ref 1), the original type consisted of a cartridge consisting of AN 91.5 & MNN (Mononitronaphthalene) 8.5%, loose in the center and compressed on the outside. It is not stated when such cartridge was introduced

Accdg to Gody (Ref 1a, pp 589 & 591), Favier expls are mixts of Amm or Na nitrate with MNN or DNN (Dinitronaphthalene), invented in 1888 in Belgium

Accdg to Marshall(Ref 2), Favier introduced his expls in 1884 into industry, especially in coal mining, because they possessed a low temp of expln and contained sufficient oxygen for complete combustion of C to CO_2, thus developing no poisonous CO

Accdg to Molina (Ref 3), the following Favier expl was introduced in 1885 in Belgium: AN 91.5 & MNN 8.5%, which is identical with compn listed in Gody (Ref 1, p 296)

Accdg to Gody (Ref 1a, p 593), the Société Belge des Explosifs Favier, at Vilvorde (Trois Fontaines) manufd before WWI three types of Favier expls:

No 1 (Antigrisou Favier) with temp of expln 2000°C contained AN 87.6 & DNN 12.4%

No 2 (Antigrisou Favier) with temp of expln 1878°C consisted of No 1 to which 8.0 parts NaCl was added

No 3(Antigrisou Favier) with temp of expln 1400°C contained AN 77.6, TNN 2.4 & Amm chloride 20.0%

Other Belg Favier expls are listed by Gody on p 712. They are *Favier II:* AN 80.9, DNN 11.7 & Amm chloride 7.4% and *Favier IV:* AN 95.5 & DNN 4.5%

Favier expls were introduced in France and the following "Explosifs de genre Favier" were manufd before WWI by the Société Française des Poudres de Sûreté: a) *Poudre Favier n°1A:* AN 88 & DNN 12%; b) *n°1B:*AN 67, Na nitrate 18 & MNN 15%; c) *n°2:* AN 44, Na nitrate 37.5 & MNN 18.5%; d) *n°3:* Na nitrate 75 & MNN 25%; e) *Grisounine roche:* AN 91.5 & DNN 8.5% and f) *Grisounine couche:* AN 95.5 & TNN 4.5% (Ref 1a, p 594). (Couche: max temp 1500°, permissible in coal seams; roche: max temp

Table F1

French Poudres Favier

Components	Designations								
	0	0a	0b	1a	1b	1c	3	4	5
AN	78.7	88.5	87.5	95	91.5	87.4	15	90	86.5
K nitrate	–	5	–	–	–	–	–	5	5
Na nitrate	–	–	–	–	–	–	58	–	–
DNN	–	–	–	–	8.5	12.6	–	–	8.5
TNN	–	–	–	5	–	–	27	5	–
TNT	21.3	6.5	12.5	–	–	–	–	–	–

Note: Poudres 0a and 0b were known as *Grisoutolite N9a* and *Grisoutolite N0b* (Ref 8)

Table F2

Designation	Components				
	AN	KNO_3	DNN	TNN	NH_4Cl
Grisou-naphalite couche	95	–	–	5	–
Grisou-naphthalite sâlpetrée	90	5	–	5	–
Grisou-naphthalite roche sâlpetrée	86.5	5	8.5	–	–
Poudre Favier pour mines nongrisouteuses	87.5	–	12.5	–	–
Favier belge antigrisouteux	81	–	6	–	13
Baelénite	93 and TNT 7%				

1900°, permissible in accompanying rock)

Vennin, Burlot & Lécorché (Ref 5, p 527) list nine French "Poudres Favier", also known as "Grisounites", "Poudres de sûreté" & "Naphthalites". Same expls manufd by les Usines de l'État (Poudrerie d'Esquerdes, Vonges, Saint Chamas & Sevran-Livry) were known as "Explosifs type N". Table F1 lists these expls

Pepin Lehalleur (Ref 6, p 352) gives compositions of the following Explosifs Favier manufd by les Usines de l'État as of 1935 (See Table F2)

Accdg to Marshall 3 (Ref 4, pp 118–19) a number of Favier type explosives have passed the Buxton Test for British "permitted" explosives. They all contained a considerable proportion of NaCl. He lists among such expls: *Ammonites No 1 & No 2, Bellite No 2, Celmonite, Gathurst Powder, Haswelite, Hawkites, Norsabite, Roburite and Tuthilite*

In other British Favier type expls NaCl was replaced by Amm chloride and a considerable proportion of potassium nitrate was introduced so that KCl could form on expln. They included *Denaby Powder, Kentite* and *Tolamex*

Davis (Ref 7, pp 350–52) describes the same Favier type expls as did Pepin-Lehalleur but he includes also *Grisou-tétrylite couche* and a number of *Grisou-dynamites* or *Grisoutines*. All of these expls are described here under "Grisou Explosifs"

Refs: 1) Daniel (1902), 295–97 1a) Gody (1907), 589, 591, 593, 594 & 712 2) Marshall 1 (1917), 388 & 390 3) Molina (1930), 337 4) Marshall 3 (1932), 118–19 5) Vennin, Burlot & Lécorché (1932), 527 6) Pepin Lehalleur (1935), 352 7) Davis (1943), 350–52 8) Clift & Fedoroff 2 (1943), p F1 (A number of Favier and Favier-type expls are listed)

Favorit. One of the names for *Koronit*, which was a Ger expl used during WWI, as supplement to nitro compds which were in short supply. Compn given in Marshall (Ref 2) came from Ref 1. It was K and/or Na chlorate 70–80, Nitro-derivs of toluene and/or diphenylamine 1–5, solid hydrocarbons and/or oils or fats 3–5 and NG 2–6%

Refs: 1) Marshall, Dict of Expls (1920), 55 2) Marshall 3 (1932), 112

FAX (Fuel-Air Explosives). See FAE and FAX

F-Bomb. Brit for Fragmentation Bomb

F.E.-1, F.E.-2 and F.E.-3. See Explosivos F.E.-1, F.E.-2 and F.E.-3 in this Vol and in Vivas, Feigenspan & Ladreda 2 (1946), p 384

Federal Fireworks Regulations. New regulations list all proposed fireworks devices permitted by HEW (the Dept of Health, Education and Welfare). Modifications include banning of bottle rockets and firecrackers, requiring safety fusing on all articles which employ fusing, providing for minimum wall thickness on tubular articles and standardizing labeling requirements to alert parents to the potential danger

Refs: 1) Federal Register, Bol **38**, No 94, Part 191d, pp 12880–84(1973) 2) G. Cohn, Edit, Expls&Pyrots **6**(8), 1973

Fedoroff, Basil T. (1891–). Russian born scientist & engineer (Merv, Russian Turkestan) who became an American citizen in 1927. He was educated before WWI at the Imperial Tomsk Institute of Technology (Chem Engr) & University of Paris, Sorbonne (Ingénieur Docteur, 1940). From 1941 he worked in the USA in the field of expls & proplnts in private industries and at Picatinny Arsenal where he was the author of a number of technical reports (PATR's) & lectures. His most important publications include: "A Manual for Explosives Laboratories", Lefax, Philadelphia, Pa, 4 Vols (1942–46) in collaboration with G.D. Clift; "Dictionary of Russian Ammunition and Weapons", PATR **2145**(1955); "Dictionary of Explosives, Ammunition and Weapons (German Section), PATR **2510**(1958); and as senior author of the "Encyclopedia of Explosives and Related Items", PATR **2700**, Vols 1–6 (1960–73). Dr Fedoroff is a unique repository of historical facts and is an internationally known expert in explosives, propellants & pyrotechnics

Ref: Oliver E. Sheffield & Gunther Cohn (1973)

Feeders and Feeding Mechanisms are devices intended for continuous and automatic carrying of materials to feed various units of a plant. Liquids and gaseous materials are usually fed by pumps, fans and pressure vessels. Solids, however, vary so greatly in character that they need special equipment. There are many types of feeders for solid materials. Most of them are described briefly by R.W. Hyde in Perry's "Chemical Engineers' Handbook", McGraw-Hill, NY, 3rd edit (1950), pp 1370–76 and in 4th edit (1963), 7–32ff

Fehleisen (Poudre de) or **Haloxyline.** An older powder: saltpeter 77.59, sawdust 15.52, charcoal 5.17 & K ferrocyanide 1.72%

Ref: Gody (1907), 173 (Compn is given in parts: saltpeter 45, sawdust 9, charcoal 3 & K ferrocyanide 1ps)

Fehling, Herman von (1812–1886). A Ger chemist known as an investigator of org compds, and of analytical methods. He proposed a reagent, known as *Fehling's Solution*, still serving for lab detection and determination of reducing sugars (Ref 1)

The Association of Official Agricultural Chemists (USA) and most other organizations use at the present time the Soxhlet modification of Fehling's Reagent, which is conducted in the following manner: Prepare No 1 soln by dissolving 34.639g $CuSO_4.5H_2O$ in distd water and dilute to 500ml; prepare No 2 soln by dissolving 173g of Rochelle salt ($KNaC_4H_4O_6.4H_2O$) with 50g Na hydroxide in w and dilute to 500ml. Allow the solns to stand for 2 days and then filter thru prepd asbestos (Ref 3). Standardize the reagent against a soln of dextrose of known strength. As a practical application of F's soln may be cited detn of glucose and sugar in glycerin used in manuf of NG

Procedure: Mix equal volumes of No 1 & No 2 solns in amts sufficient to fill half of a test tube and fill the remaining half with an equal volume of glycerin. Stopper the tube so that no air remains under stopper and let it stand for 12 hrs. A reddish ppt indicates the presence of reducing agents which will interfere in testing for glucose or sugar, unless the ppt is removed by filtration

If the previous test is negative, transfer the contents of the tube (without filtering) into a larger tube, add more F's soln and heat the liquid for a few minutes. The resulting red ppt of Cu_2O (if present) is filtered, washed with w, ignited and weighed as CuO. Compare the wt with that of "standard" obtd by treating a dextrose soln of known concn

For determination of sugar (saccharose) in glycerin, dilute a known vol with two vols of w and, after acidifying it with few drops of HCl, heat just to boiling and nearly neutralize with Na_2CO_3. Add an excess of F's soln, heat for few mins and treat the resulting red ppt as described above

Refs: 1) Hackh's (1944), 333-R 2) G.D. Clift & B.F. Fedoroff, "A Manual for Explosives Laboratories", Lefax, Inc, Phila, Vol 1, Chap XI, pp 3–4(1942) 3) CondChemDict (1961), 485

Feigl, Fritz (1891–). Austrian analytical chemist; educ Poly Inst of Vienna (BChE, 1914; DSc, 1920); assoc since 1940 with Ministry of Agric, Brazil; developed spot test methods for qualitative analysis requiring min amts of sample and reagent and very simple apparatus

Refs: 1) Anon, C&EN **27**, 1997 (1949) 2) V. Anger, ZAnalChem **184**, 1 (1961)(A bibliography with picture)

Fehleisen & André patented ca 1866 Black Powders contg K ferricyanide in lieu of sulfur. Another variety contd K ferrocyanide which was patented by Fehleisen & Bleckmann

Ref: Daniel, Dict (1902), 298 & 367

Felhoen Powder – patented in England in 1879 consisted of 90p of Black Powder with 10p of a product obtained on nitration of naphthalene (1p) with 4parts of nitric acid (d 1.4). The nitrated product consisted of mononitronaphthalene with a small amount of the dinitro-compd

Ref: Daniel (1902), 298

Felixdorf Factory Ammonals (Austrian). See Vol **1**, p A289 (Table)

Felixites. Brit sporting bulk powders. Some of the older types are listed inRef 1 and some of the newer ones in Ref 2.

Examples: 1) *Felixite 1906* (fibrous, 42 grain bulk): NC(insol) 40.5, NC(sol) 20.5, metallic nitrates 30, nitrohydrocarbon 5.0, vaseline 2.7, moisture 1.3%

2) *Felixite NE 1912* (fibrous, 36 gr bulk): NC(insol) 50.0, NC(sol) 25.8, metallic nitrates 12.0, nitrohydrocarbon 7.0, vaseline 3.5, moisture 1.7

3) *Neonite 1907* (gelatinized, 30 gr bulk): NC(insol) 73.0, NC(sol) 9.0, metallic nitrates 10.5, vaseline 5.9, moisture 1.6

Refs: 1) Marshall **1** (1917), 236 2) Ibid **3** (1932), 96

Fenchone (1,3,3-Trimethyl-2-norcamphanone), $C_{10}H_{16}O$; mw 152.23; colorless oil with camphor-like odor, sp gr 0.9465 at 19°, fr p 5–6°, bp 193–95°; insol in w, v sol in alc or eth. This ketone is found as dextrofenchone in oil of fennel, and as levofenchone in oil of tuja. It is a solvent for NC forming a molecular compd in solns and has been used as moderator and gelatinizing agent in single-base or double-base proplnts

Refs: 1) Beil **7**, 96 2) C. Claessen, USP 979431 (1910) 3) T. Tomonari et al, ZPhysikChem **B17**, 241 (1932) & CA **26**, 4229 (1932) 4) CondChemDict (1961), 485-R

Fenian Fire (Feu liquide in Fr). Incendiary compn consisting of a soln of yellow phosphorus in CS_2. After evapn of the solv, phosphorus catches fire and ignites the surrounding flammable materials. Darapski proposed charging incendiary projectiles with the soln consisting of yellow phosphorus (3p) and CS_2 (1.3p). Inside this soln was placed a leather bag contg petr oil with other combustible materials

Ref: Daniel (1902), 153

Fenices (Brit & Fr). A series of Dynamites patented in 1899: a) NG 25, K nitrate 34, Na nitrate 1 & sawdust 40% b) NG 25, Na nitrate 35 & rye flour 40% c) NG 30, Na nitrate 32 & sawdust 38% d) NG 30, Na nitrate 30 & rye flour 40%

Ref: Giua, Trattato **VI**(1)(1959), 386

Fenton's Powders. Explosive mixtures of K chlorate, K ferrocyanide and sugar patented in England in 1873 (Ref 1). Pérez Ara (Ref 3) gives for Pólvora de Fenton: $KClO_3$ 66, $K_4Fe(CN)_6$ 17 & sugar 17%, while Giua (Ref 4) gives for Polvere Fenton (1873): $KClO_3$ 50, $K_4Fe(CN)_6$ 25 & sugar 25%. The compn given by Giua is identical with that given in Davis (Ref 2) and in Vol 1 of Encycl, p A507-L for Augendre Powder (White Powder), proposed in 1849 in France, and used later in other countries

Refs: 1) Daniel (1902), 298 1a) Gody (1907), p 264 (Poudre de Fenton) 2) Davis (1943), 358 3) Pérez Ara (1945), 212 4) Giua, Trattato, **VI**(1)(1959), 392-R

"Ferdinand". Ger tank (Panzer) of WWII, known as *JgdPz VI(P), Tiger Porsche* or *SdKfz 184.* It was Tiger 1 (H) of Henschel converted by Porsche into self-propelled motor carriage. It was equipped with one MG and one long-barreled 88mm Gun (8.8cm KwK 36). It was superseded by "Elefant" (See in this Vol)

Refs: 1) G.B. Jarrett, "Achtung Panzer", "The Story of German Tanks in WWII", Great Oaks, RD 1, Aberdeen, Md (1948) 2) Fedoroff et al, PATR **2510**(1958), p Ger 125-R, under PANZER

Fergusonite. A natural inorg substance discovered among the feldspars of Norway, but it is found also in Sweden, USA and Africa. It is an oxide of ytrium, erbium, niobium & tantalum – $(Y, Er, Nb, Ta)O_4$, contg small amounts of helium, cerium, uranium, etc. It is a gray, brown or black solid, sp gr 5.6–5.8 and hardness 5.5–6.5 (Ref 2). Accdg to Daniel (Ref 1), when fergusonite is heated, it suddenly flashes, developing a very high temp and pressure due to the evoln of a large vol of He. Its heat of expln is ca 800cal/g

Refs: Daniel (1902), 298 2) CondChemDict (1961), 486-R

Fermentation. Fermentation is defined (Ref 3) as the production of chemicals by a series of enzyme catalyzed reactions with bacteria, yeasts, or molds under aerobic or anaerobic conditions. At present, fermentation is used to produce complex molecules not easily synthesized such as penicillin and other antibiotics, vitamin B_{12}, and enzymes. Formerly, glycerine (See Fermentol), acetone, butanol, and citric & lactic acids were some of the chemicals produced by fermentation process. Synthesis is now a more economical route to these materials (See also Refs 1 & 2)

Refs: 1) P.A. Wells & G.E. Ward, IEC **31**, 172–77(1939) 2) H.E. Silcox & S.B. Lee, IEC **40**, 1602–08(1948) (First of a series of articles on fermentation) 3) Kirk & Othmer, 2nd edit **8**(1965), 871–80 (R.G. Anderson, Fermentation)

Fermentol (also *Protol Glycerine* or *Protoglycerine).* A glycerine produced in Germany from sugar by a fermentation process. Trimethylene glycol in the product evolves more heat than glycerine on nitration, is more soluble in the mixed acid, and thus contributes to fume-offs

Refs: 1) Naoúm, NG(1928), pp 31, 229 2) Marshall **3**(1932), p 42

Ferric Azide. See under Azides in Vol 1, p A543

Ferric Azide, Basic. See Vol 1, p A543-R

Ferric Ferrocyanide or Prussian Blue, $Fe_4[Fe(CN)_6]_3$; mw 859.29, dk blue crysts, mp – dec; insol in cold w, alc or eth; sol in

HCl & concd sulfuric acid. Prepd as described in Ref 2 under Iron blues. Used as an ingredient of some expl compns
Refs: 1) Lange (1961), No 815 2) CondChemDict (1961), 615 (Iron blues) 3) Kirk & Othmer **12**(1967), 33–35 (under Iron Compounds by R.S. Casey & J.R. Doyle)

Ferric Nitrate (Iron Nitrate), $Fe(NO_3)_3.9H_2O$; mw 404.02, lt viol monocl delq crysts, mp 47°, dec at 100°; v sol in w & alc. Can be prepd by the action of concd nitric acid on scrap iron or on iron oxide, followed by crystn. It is a strong oxidizing agent and is considered to be dangerous as a fire hazard. Used in analytical chemistry
Refs: 1) Lange (1961), No 822.1 2) CondChem Dict (1961), 488-R & 489-L 3) Kirk & Othmer **12**(1967), 37–38 (under Iron Compounds)

Ferric Oxide or Red Iron Trioxide (Hematite), Fe_2O_3, mw 159.70, red or blk trigonal crysts, sp gr 5.12, mp 1560°(dec); insol in w, sol in HCl & other acids; found in nature as hematite and is a by-product in some industries. Can be prepd by dehydrating ferric hydroxide or calcining ferrous oxalate or sulfate. Used in magnetic tapes in electronics and as an ingredient of some expl compns
Refs: 1) Lange (1961), No 824 2) CondChemDict (1961), 489 3) Kirk & Othmer **12** (1967), 39 (under Iron Compounds)

Ferric Picrate. See under Picrates

Ferric Triazide. Same as Ferric Azide, Vol **1**, p A543

Ferricyanides. See the cation (potassium, sodium, etc) for particular ferricyanides

Ferri-ferrous Oxide or Magnetite, also known as *Iron Oxide, Black* or *Ferroso-ferric Oxide*, Fe_3O_4; mw 231.55, black cubic crysts or amorphous powder, sp gr 5.0, mp 1538°(dec); insol in w, alc or eth; sol in acids; occurs in nature as mineral magnetite. Can be prepd in pure state by dehydrating pptd hydrated ferric oxide, followed by reduction with hydrogen. Used as an ingredient of some expl compns and in *ferrite* (qv) for electronic industry
Refs: 1) Lange (1961), No 835 2) CondChemDict (1961), 617-L (Iron Oxide, Black) 3) Kirk & Othmer **12**(1967), 38 (under Iron Compounds)

Ferrifracteur. A safety expl contg AN 90 & DNB 10%
Ref: Daniel (1902), 299

Ferrite. Iron which, in pig iron or steel, has not combined with carbon to form cementite (Fe_3C). It exists in α, β, γ and δ forms, which vary in magnetism and ability to dissolve cementite. Name also applied to compd $NaFeO_2$ (called Na ferrite), to ferromagnetic oxides having a definite cryst structure (spinels) and the formula $M^{++}Fe_2^{+++}O_4$ of which the divalent metal may be Fe, Ni, Zn, or Mn. The magnetic props vary accdg to the divalent atom present, and ferrites are now tailored for their desired effect, as Ni-Al ferrite;
$Ni^{++}_{0.86}Cu^{++}_{0.1}Co^{++}_{0.02}Al^{+++}_{0.3}Fe^{+++}_{1.7}O_4$
Used in missile guidance systems; in electronics as rectifiers, on memory or record tapes; for permanent magnets; also in radar, radio, television and computers
Refs: 1) CondChemDict (1961), 491-L 2) Kirk & Othmer, not found

Ferro-Alloys, Chrome Metal, and Spiegeleisen are covered by Federal Specification **QQ-F-145**, April 30, 1964, with Amendment 1, January 18, 1967. The following alloys are covered by this spec: Ferrochromium, Chrome Metal, Ferromanganese, Ferromolybdenum, Ferrosilicon, Ferrotitanium, Ferrovanadium and Spiegeleisen

Their compositions and tests are given in the spec. They are intended to be used as alloying additives in the melting and preparation of ferrous and nonferrous alloys
Ref: CondChemDict (1961), 491-L

Ferroboron. A ferro-alloy averaging 16.2% boron used as hardening agent in special steels. It also is an efficient deoxidizer. Boron steel is used in controlling the operating rate of the uranium-graphite piles used to produce plutonium
Refs: 1) CondChemDict (1961), 491-R 2) Kirk & Othmer, **3** (1964), 605 -08 (under Boron and Boron Alloys by F.E. Bacon)

Ferrocene or Diclopentadienyl-iron, $(C_5H_5)_2Fe$. A coordination compd of ferrous iron and dicyclopentadiene in which the org portions have typically aromatic chem props. It is an orn cryst compd with camphor-like odor, mp 173–74°, resists pyrolysis at 400° and is resistznt to UV light. Insol in w; sl sol in benz, eth & petr eth. Can be prepd from ferrous chloride and cyclopentadiene sodium. Used as additive to jet fuels and furnace oils to improve efficiency of combstn and to eliminate smoke; antiknock additive for gasoline; high temp lubricant; curing agent for rubber & silicone resins; intermediate for high temp polymers and UV absorber. It was suggested for coating missiles and satellites
Refs: 1) Beil **5**, 495 (Dicyclopentadiene) 2) CondChemDict (1961), 491-L & R (Ferrocene) 3) Kirk & Othmer **6** (1965), 696 (under Cyclopentadiene)

Ferrochromium (Ferrochrome). An alloy composed principally of Fe & Cr used as a means of adding Cr to steels, such as in manuf of stainless steels. The alloy is available in several classifications and grades, generally contg betw 60 & 70% Cr. Stainless steels contain 10 to 25% Cr and some of them contain Ni. Three classes of such steels: austenitic, ferritic and martensitic are described in Ref 1, p 1074. There also are many types of **Enduro** (Trade Mark of Republic Steel Corp, Cleveland, Ohio) stainless and heat resistant steels. They are described in Ref 1, pp 440 & 441-L
Refs: 1) CondChemDict (1961), 441 (Enduro); 491 (Ferrochromium); and 1074-L (Steel, Stainless) 2) Kirk & Othmer, not found

Ferroconcrete. One of the names for concrete reinforced by steel in various forms, usually rods
Refs: 1) CondChemDict (1961), 295 (Concrete); 491 (Ferroconcrete) 2) PATR **2700**, Vol **2** (1962), pp C125-R & C126-L (Cement and Concrete in Ordnance) 3) Kirk & Othmer, not found

Ferrocyanides. See cation (potassium, sodium, etc) for particular cyanides. Accdg to Marshall **1** (1917), K ferrocyanide has been used as a constituent of blasting expls

Ferrosilicons are compds prepd by fusing iron and silica in an electric furnace at high temps. Some of the high silicon varieties, particularly 50/50 compd, are expl and are dangerous to transport and to handle. FeSi is of unknown toxicity. Moisture, steam, or water decompose it and impurities liberate phosphine and arsine with water. Expl and fire hazards are considered moderate and arise from the reaction with water. For shipping purposes FeSi with Si 30–70% is considered a flammable solid and must carry yellow labels. If the Si is less than 30 or more than 70%, the material is not considered hazardous

J. Sato (Ref 10) reported FeSi decreased the rate of deton of RDX and TNT. Médard (Ref 4) reported that CUP of several expls increased slightly with 10% FeSi: CUP of PA increased from 100 to 102%, TNT 94.0 to 94.5, PETN 146.5 to 148 and RDX 134.5 to 141%. Sartorius (Ref 5) reported slight increases in Trauzl Tests of the same four expls with 10% FeSi. Refs 6, 7, 8, 9, 10 and 11 give formulations from recent patents where FeSi was added. In general, 10% FeSi gave slight improvements in props of the comps in which it was included
Refs: 1) Anon, ElectrochemMetInd **7**, 64 (1909) & CA **3**, 1087 (1909) 2) Anon, EngrgNews **83**, 300 (1910) & CA **4**, 1678 (1910) 3) S.M. Copeman, ChemNews **102**, 127 (1910) & CA **4**, 304 (1910) 4) L. Médard, MAF **22**, 601 (1948) 5) R. Sartorius, MP **34**, 208 (1952) 6) C.O. Davis et al, USP 2752848 (1956) & CA **50**, 13444 (1956) 6a) CondChemDict (1961),

492-R 7) R. Mecir et al, CzechP 89625 (1959) & CA **55**, 27890(1961) 8) W.F. Kirst, USP 3026331(1962) & CA **56**, 14522(1962) 9) F.A. Loving, Jr, USP 3092528(1963) & CA **59**, 6192(1963) 10) J. Sato, Kôgyô-KayashiKyôkaishi **27**(3), 180–82(1966) & CA **66**, 97073(1967) 11) Sax (1968), 774 12) H.G. Knight, USP 3528841(1970) & CA **73**, 79084(1970)

Ferrous Acetylide. See Iron Acetylide in Vol **1**, p A76-R under ACETYLIDES AND CARBIDES

Ferrous Ammonium Sulfate or Mohr's Salt. $FeSO_4.(NH_4)_2SO_4.6H_2O$; mw 392.16, blue-grn monocl crysts, sp gr 1.864, mp – dec; sol in w, insol in alc. Can be prepd by mixing aq solns of equivalent wts of Amm and ferrous sulfates, followed by evapn and subsequent crystn. Used for $-NO_3$ detn in inorg & org nitrates, as described under "Ferrous Sulfate and Ferrous Ammonium Sulfate in Determination of Nitrates"
Refs: 1) Lange (1961), No 839 2) CondChemDict (1961), 493-R 3) Kirk & Othmer, not found

Ferrous Azide. See Vol **1**, p A543-R

Ferrous Carbide. See Iron Carbide in Vol **1**, p A76-R under ACETYLIDES AND CARBIDES

Ferrous Oxide (Iron Monoxide), FeO; mw 71.85, blk pdr, sp gr 5.7, mp 1420°; insol in w, sol in acids. Can be prepd by heating ferrous oxalate under the hood, while avoiding inhalation of toxic CO evolved. Some ferric oxide is present as impurity. Used as ingredient of some expls
Refs: 1) Lange (1961), No 859 2) CondChemDict (1961), 494-R 3) Kirk & Othmer **12** (1967), 38 (under Iron Compounds by R.S. Casey & J.R. Doyle)

Ferrous Picrate. See under Picrates

Ferrous Sulfate *(Iron Sulfate* or *Green Vitriol)*, $FeSO_4.7H_2O$; mw 278.03, blue-grn monocl crysts, sp gr 1.899 at 18.8°, mp 64°, $-7H_2O$ at 300°; sol in w, insol in alc. Can be prepd by the action of dil sulfuric acid on iron or by other methods. Used in lab for detn of inorg or org nitrates (See next item)
Refs: 1) Lange (1961), No 865 2) CondChemDict (1961), 494-R 3) Kirk & Othmer **12**(1967), 40–42 (under Iron Compounds)

Ferrous Sulfate and Ferrous Ammonium Sulfate in Determination of Nitrates.
Historical: The first mention of a method was made by Grossart in 1847 (Ref 1), who titrated the nitrates in boiling sulfuric acid with a $FeSO_4$ soln using ferricyanide as an outside indicator. Mohr (Ref 2) described in 1861 a method in which the sample was dissolved in dilute sulfuric acid and titrated at 70–80° with ferrous sulfate soln until the appearance of a brownish coloration. This method was condemned in 1862 by Fresenius and by Eder in 1877. The method was forgotten until Devender proposed in 1899 to carry the reaction over mercury with exclusion of air. The process was awkward to handle and was not adopted. A new impetus was given to the method by Bowman and Scott (Ref 3), who proposed a method which is still successfully used (with minor modifications) in many laboratory tests. The method is expanded now to determination of NO_3^- in organic esters, such as NG, NC, etc

Because the end point (formation of a permanent brownish coloration) is not very sharp, the back titration method (Ref 4) was used successfully by Dr Fedoroff at the Keystone Ordn Works, Meadville, Pa and improved at PicArsn (Ref 9a). More recently, an electrometric method for detection of the end point was proposed (Refs 5, 6, 7, 8 and 11; Refs 6 & 7 are discussions of instrumental design and not primarily about $FeSO_4$ to detn nitrate). The method developed at PicArsn for detn of N in NC is described in detail in Ref 18

In the back titration method an excess of $FeSO_4$ is added to the soln of the nitrate and back titrated with $K_2Cr_2O_7$ (Ref 4). In addn to the use of $K_2Cr_2O_7$, Kolthoff et al

adopted the use of Amm molybdate to catylize the rather slow reduction of NO_3^- by Fe^{++}

In 1956, Fréjacques and LeClercq (Ref 12) suggested the use of ferrous ammonium sulfate (qv) because the Fe^{++} in it was not as subject to oxidation by air

A colorimetric method based on the violet color produced by ferrous sulfate in sulfuric acid in the presence of NO_3 was announced by English in 1947 (Ref 9) and applied to the determination of NA and nitrosylsulfuric acid (NSA) in spent mixed acid. Since then a number of papers (Refs 13, 14, 15, 16 and 17) have extended the method to NO_3^-, organic nitrates and RDX–HMX mixtures

Analytical Methods: The visual determination of the endpoint (appearance of a permanent brown color) in the dead-stop titrimetric method is reported accurate to 0.03ml (Ref 3) and was used recently by Fréjacques and LeClercq (Ref 12) for analysis of Pentolites, Tetryls, NGu, NG prepns, and Nitroethane-EGDN expls. The endpoint has been criticized as difficult to see, and if problems arise, electrometric methods are available for detection (Refs 6, 7, and 8). The back titration method (Ref 4) was used by Leith (Ref 10) for fertilizers and Potier (Ref 11) for nitrate in water and sulfuric and nitric acids

Any of the methods mentioned are applicable to NA or to inorg NO_3^-. Application to org nitrates depends on hydrolysis of the org compd by the sulfuric acid in which the ferrous sulfate reagent is prepd or the sample dissolved to liberate the NO_3^- for reaction. The residues of the organic portion of the molecule remaining could be oxidized by the potassium dichromate used in the back titration method and so introduce errors. The dead stop method or the colorimetric method (below) might be preferred in such cases

The references cited for the colorimetric method differ primarily in sample treatment needed to get the sample ready for analysis. The actual determination consists of adding a soln of ferrous sulfate in sulfuric acid and measuring the absorption at 510–515 nanometers. A period of 30–90 mins is needed for development of the color

English (Ref 9) reported the development of the method for the determination of NA and nitrosylsulfuric acid (NSA) in spent MA. Both NA and NSA give the color reaction so the total is determined, the NSA destroyed with sulfamic acid, and the NA alone detd. NSA is then the difference. The adaptation of the method to NC was described in Ref 13. The sample was dissolved in wwter or acetone for the determination. Interferences reported are: NO_2^- and $S_2O_3^=$; while $CrO_4^=$, $Cr_2O_7^=$, $SO_4^=$, $PO_4^{\equiv}$, ClO_3^-, $SO_3^=$, OAc^-, and Hal^- did not interfere. PETN, HNMnt, NG, and Inositol $(NO_3)_6$ in pharmaceutical tablets were detd (Ref 15) by extracting the org nitrate with $HCCl_3$ (or acet for PETN) from the excipient (lactose or mannitol), evaporating to dryness, and taking up the residue in glac acet ac. Na_2SO_3 was used as an accelerator in the color development stage. NMe, NB, MNt and Nitrourea gave no color

In two papers (Refs 15 & 16), Lacetti, Semel and Roth reported use and development of the method for organic nitrates of Ordnance interest and extended the method to nitramines used in proplnt and HE compns. The reaction was not specific for $-O-NO_2$ since $-N-NO_2$ was found to give the same color. NG, PETN, TEGDN, Tetryl, NGu all gave the same straight line when absorbance was plotted against concentration, permitting one calibration curve to be used for all. RDX and HMX also gave straight lines but of different slopes from each other and from the other materials. Since the RDX and HMX curves differ, a weighed sample of a mixture will have an absorbance between the values for the same weight of either of the pure compounds, thus permitting detn of one of them in the presence of the other. Norwitz (Ref 17) detd inorg nitrate in proplnts by extracting the NC with acet, filtering to remove the undissolved nitrate and dissolving the residue in water. Common constituents of proplnts did not interfere, but oxidizing agents caused high results and NO_2^- interfered

Refs: 1) Grossart, CR **1**, 21 (1847) 2) Mohr, Dingler's PolytechnischesJ, **160**, 219 (1861) 3) F.C. Bowman & W.W. Scott, IEC **7**, 766 (1915) 4) I.M. Kolthoff et al, JACS **55**, 1454 (1933) (Scott's StdMethChemAnal, 5th edit (1939), p 644 (includes the proc for this method) 5) W.D. Treadwell & H. Vontobel,

HelvChimActa **20**, 573(1937) 6) J.R. Gay, IEC AnalEd **11**, 383(1938) 7) E.J. Serfass, IEC AnalEd **12**, 536(1940) 8) C.D. McKinney, Jr et al, IECAnalEd **19**, 1041(1947) 9) F.L. English, AnalChem **19**, 850(1947) 9a) B.T. Fedoroff, PicArsnChemLab Rept **122871**(1948) (GenLab-File No 1123) 10) W. Leithe, AnalChem **20**, 1082(1948) 11) A. Potier, MP **32**, 489(1950) 12) C. Fréjacques & M. LeClercq, MP **38**, 39 (1956) & CA **51**, 11717(1957) 13) M.H. Swann & M.L. Adams, AnalChem **28**, 1630 (1956) 14) F.J. Bandelin & R.E. Pankratz, AnalChem **30**, 1435(1958) 15) M.A. Laccetti, M. Semel & M. Roth, AnalChem **31**, 1049(1959) 16) S. Semel, M.A. Laccetti & M. Roth, AnalChem **31**, 1050(1959) 17) G. Norwitz, AnalChem **34**, 337(1962) 18) B.T. Fedoroff & O.E. Sheffield, PATR **2700**, Vol **2**(1962), pp C122-R & C123-L & R (Detailed description of ferrous sulfate or ferrous ammonium sulfate for N dem in NC, developed in 1948 at PicArsn and originally described in Ref 9a)

Ferrous Sulfide (Iron Protosulfide), FeS; mw 87.92, blk hex metallic solid, sp gr 4.84, mp 1193°, bp decomp; insol in w, sol in acids. Can be prepd by fusing iron with equiv amt of sulfur. Used for generating hydrogen sulfide
Refs: 1) Lange (1961), No 866 2) CondChemDict (1961), 495-L 3) Kirk & Othmer **12**(1967), 42 (under Iron Compounds) 4) US Specification MIL-F-279B (May 1969) (Ferrous Sulfide, Technical)

Fertilizer Distributed by Explosives. W.O. Snelling obtained 3 patents (USP 1274343, 1308453, 1316005) for methods of distributing fertilizer by expls. The fertilizer in large lumps is mixed with the expl or packed in a container around a core of expl. Deton breaks up the lumps and distributes the expl thru the ground

Fertilizer Grade Ammonium Nitrate. See FGAN

Fertilizer Recovered from Explosives. AN in unneeded expls is recovered by leaching with water, adding peat to the solution and drying (Refs 2 & 3). When the expl contains perchlorate, the water is replaced by KCl soln which eliminates the perchlorate (Ref 1). The expl may also be agitated at room or elevated temp with hydroxides of alkali or alkaline earth metals (pref calcium hydroxide) with or without application of pressure (Ref 4) [See also Ammonium Nitrate, Fertilizer Grade (FGAN) in Vol 1 of Encycl, p A364]
Refs: 1) Dynamit AG, vorm Alfred Nobel & Co, GerP 321878(1918) & CA **15**, 1961 (1921) 2) F. Garelli, AnnAccadAgrTorino **62**, 75–80 (1919) & CA **15**, 3174(1921) 3) S. Lissone, IndustrieChim **1**, 29–30(1919) & CA **15**, 3715 (1921) 4) A Heinritzi, ItalP 433961(1948) & CA **44**, 3684(1950)

Fescolizing of Shots. A nickel plating process for ballistic mortar projectiles by Fescol, Ltd, hence the name. The useful life of the mortar and the projectiles is increased
Ref: J. Taylor and J.H. Cook, JScInst **26**, 267–8(1949)

Feu. (Fr) Fire; firing

Feu d'artifice. (Fr) Fireworks

Feu d'artifice de guerre, (Fr) Military fireworks

Feu de canon. (Fr) Artillery fire

Feu grégeois. (Fr) Greek fire

Feu grisou. (Fr) Firedamp

Feu liquide. (Fr) See Fenian fire

Feu lorrain. (Fr) See Lorrain fire

Feu prussien. (Fr) See Eigfall powder

FF-30. A std commercial ignition material for "Therm 64C" consisting of the following compn: Ti 30 & Fe_2O_3 (red pigment grade 70%
Ref: Tech Command, ArmyChemCenter, Maryland, "Development of an Incendiary Pellet", **TCR-59** (May 1950)

FGAN (Fertilizer Grade Ammonium Nitrate). See AMMONIUM NITRATE, FERTILIZER GRADE in Vol 1, pp A364-L to A367-L and also pp A359-R to A362-L

F.G. Powder. Fine grain Black Powder used in the 19th century in England as proplnt for smaller caliber cannons and as a bursting charge for shrapnel shells
Ref: Daniel (1902), p 300

FI (Figure of Insensitiveness) Test. See FI Test

Fiber or Fibre is any tough substance composed of threadlike tissue, especially when capable of being spun or woven. Fibers may be divided into animal (wool or silk), vegetable (cotton, hemp, flax, ramie, esparto, jute, sisal etc), mineral (asbestos, glass fiber) and artificial (Rayon, Nylon, Orlon, Vinyon, Saran etc)

All the vegetable fibrous materials are more or less suitable for the preparation of explosives (See Nitrocellulose, Nitrojute, Nitroramie, etc) (See also Cotton, Vol 3 p C545ff)
Refs: 1) T.M. Plitt, "Microscopic Methods Used in Identification of Commercial Fibers", USGovtPrintingOffice, Washington (1939) 2) L. Weinling, "Long Vegetable Fibers", Columbia Univ Press, NY (1947) 3) J.M. Matthews & H.R. Mauersberger, "Textile Fibers; Their Physical, Microscopical and Chemical Properties", J. Wiley, NY (1947) 4) Kirk & Othmer, Edits, "Encyclopedia of Chemical Technology", Interscience, NY, Vol 6(1951), pp 453-476; D. Himmelfarb, "Fibers" (8 refs) and 2nd Edition, 9(1966), pp 151-70, H.F. Mark & S.M. Atlas, "Fibers, Man-Made"; pp171-85, D. Himmelfarb, "Fibers, Vegetable" (9 refs)

Fickett Equation of State Based on Intermolecular Potentials. See Vol 4 of Encycl, p D277-R

Fickett-Wood Equation of State. See "Constant-β and Constant-γ Equations of State", Vol 4, pp D275-L & D275-R

Fickett-Wood-Salsburg Discussion on Equation of State Based on Intermolecular Potentials. See W. Fickett, W.W. Wood & Z.W. Salsburg, JChemPhys **27**, 1324-29 (1957)

Field Chronograph Test. See Vol 3, p C310-L, under CHRONOGRAPHS

Field Clearing with Dynamite or Other Explosives. This means the removal of stumps, boulders and rock ledges by the use of suitably placed and sized charges of Dynamite (or other explosives). Such variables as soil compressibility, hardness of the rock or wood, overall massiveness, etc make the writing of rules impossible. In general a single charge is placed in a hole under a lateral rooted stump; multiple charges are placed between the roots of a semi-tap rooted stump and fired simultaneously, electrically; tap rooted stumps may be drilled for one charge to remove only the upper portion, or dug around deeply on opposite sides for the simultaneous firing of two charges for total removal. Boulders are handled either by placing a charge underneath – snake holing, or on top covered with a layer of mud – mud-capping. Rock ledges are best handled by drilling, but the boulder methods will work too, preferably snakeholing
Refs: 1) Encycl of Explosives, PATR **2700**, Vol 1 (1960), p A113-L, Items b & c
2) Blasters' Handbook, 15th Edit (1966), pp 378-384

Fielder Explosive. A Sprengel type explosive, invented in Russia by Fielder, was prepd just before use by mixing the liquid ingredient (fuel) with a solid ingredient (oxidizer). The fuel was a mixture of NB 80 & turpentine 20%,

whereas the oxidizer was a mixture of K chlorate 70 & K permanganate 30%
Refs: 1) Daniel (1902), 300 2) Thorpe, Vol 2(1912), 421 3) Davis (1943), 355 4) Pérez Ara (1945), 231

Fieldner, Arno C., Dr (1882–1966). Pioneer US Govt research scientist and internationally known authority on coal and other fuels who died July 13, 1966 after an automobile accident. He graduated from Ohio State University and began work as a chemist at the BurMines at the time of its founding in 1910. During WWI he directed research on gas masks and adsorbents for CWS. In 1942 he was named chief of fuels and explosives services at BurMines, and in 1950, as chief fuels technologist. He retired in 1955
Ref: Anon, "Arno C. Fieldner", C&EN 44 (35), p 116 (Aug 29, 1966) (Obituary, with portrait)

Field Parachute Drop Test for Fuzes. See Vol 4, p D1093-L

Field-Proof Gun. A special shot gun for taking simultaneously the recoil and pressures at 1 inch and 6 inches from the breech. The velocity and pattern can also be taken at the same time. The gun weighs 50 lbs and is suspended 5 ft below the supports; it is fired by means of a pneumatic bulb in order not to disturb the gun. The gun and support are shown in Fig F2

The "pattern" of the shot is detd by firing at a whitewashed iron plate, generally at a range of 40 yards. The marks of the shot should be fairly evenly distributed, and about 2/3 rd of the shot should be within a circle of 30 inches diameter

The penetration of shot can be measured by firing under standard conditions at a number of pieces of cardboard placed one behind the other and counting the number of pellets that penetrate different cards
Ref: Marshall 1(1917), 332 & 333

Field Samples of Permissible Explosives. See Vol 3, p C376-R, under Coal Mining Explosives, Testing for Permissibility

Field Testing of Explosives. When unexploded ammunition is found in the field, it is usually required that it be destroyed or rendered inert. It is necessary to know how sensitive the charge is in order to know how to handle it. This can be determined if the various expl components are identified directly in the field; it is advisable to carry a magnifier.

The following general characteristics of expls sometimes help in identifying them:
1) *Appearance*, such as color, gives an indication of the approx identification: TNT (buff or brownish), Picric Acid and Explosive D (yellow), RDX and PETN (white). However, since as many other expls are brown or yellow in color, this method is not very reliable. The question is further complicated if the expl is dyed, a practice in some foreign countries
2) *Method of loading*, such as press- or cast-loaded, might give some indication as to the identity: TNT (cast), Tetryl (pressed), etc, but it is not always possible to distinguish the method of loading unless a magnifying glass is used
3) *National and functional use of the expl.* If the national origin of the expl and its functional use are considered in conjunction with the color and method of loading, it is sometimes possible to correctly identify the expl. For instance, if an American booster charge is a yellow press-loaded compound, one can be sure that it is Tetryl; white booster can be either RDX or PETN. In the latter case further identification is unnecessary because both expls are practically of the same sensitivity. As PETN is slightly more sensitive, it is safer to treat the expl as though it were PETN
4) *Sensitivity.* In order to ascertain how carefully an expl has to be treated, it is necessary to determine its sensitivity. This is especially important in the case of primary and initiating expls

The following Field Tests give an approx idea of the sensitivity:
a) Remove a few particles of the expl by the use of a long wooden or plastic spatula and

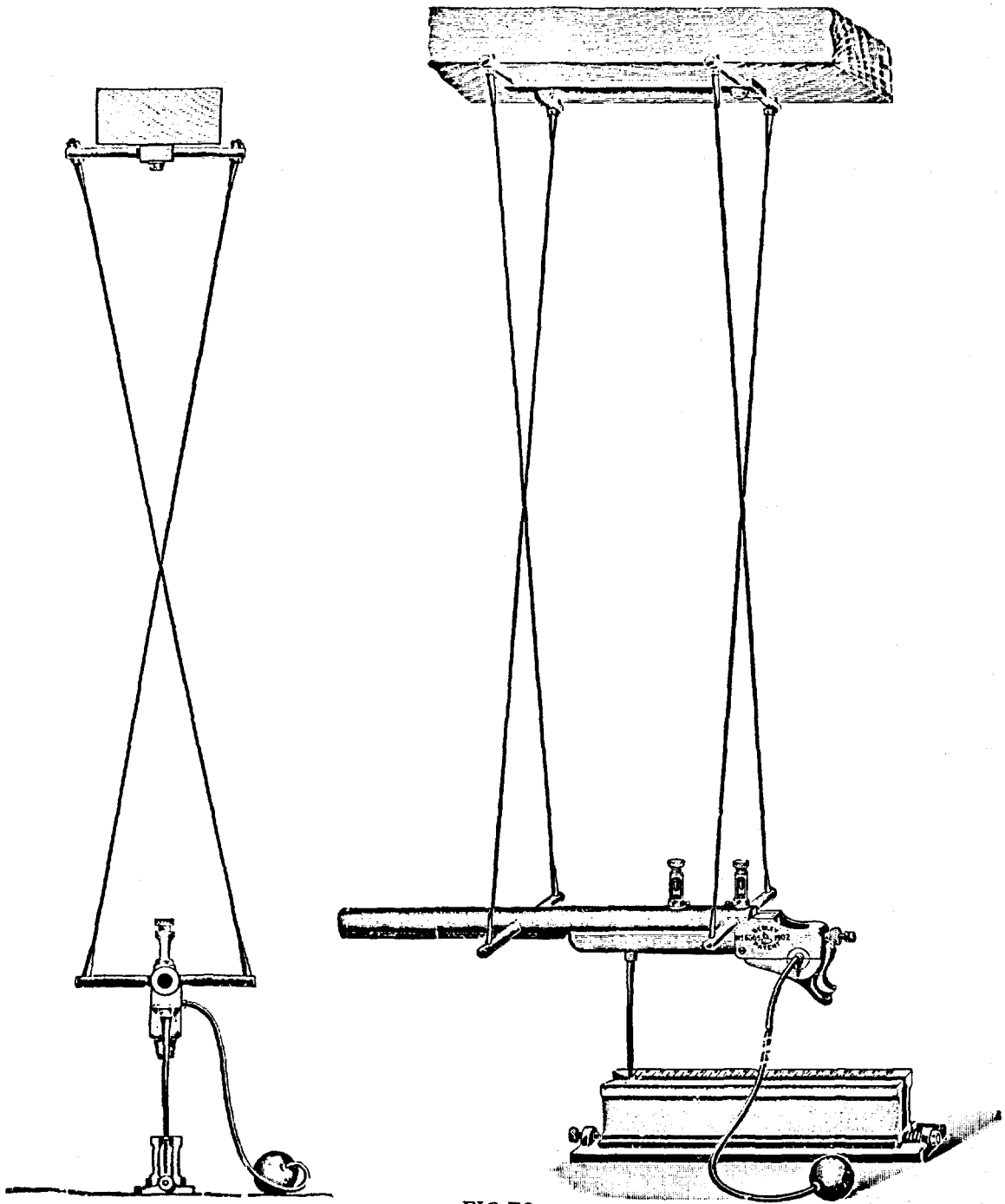


FIG F2
FIELD PROOF GUN

Table F3

Tests of Noninitiating Explosives

	Water solution or extract			Color effect of test with—		
	Color	Color of universal pH test	Color of PPT with Nessler's	Ethylenediamine	Diphenylamine	Thymol
TNT	(Insoluble)			Maroon	Colorless	
Tetryl	(Insoluble)			Red	Blue	Green.
Picric acid	Yellow	Red	(No ppt)	Orange		
Explosive "D"	Yellow		Brown	Orange		
Haleite	None		(No ppt)	None	Blue[1]	Orange.
Nitroguanidine	None		White		Blue	Green.
Ammonium nitrate	None		Brown	None	Dirty green	Green.[1]
PETN	(Insoluble)			None	Dirty green	Green.
Nitroglycerin	None		(No ppt)		Deep blue	Green.
DEGN	None		(No ppt)		Deep blue	Brown.[2]
Nitrocellulose	None				Blue	Green.
Tritonal	(Insoluble)			Maroon	Colorless	
Tetrytol	(Insoluble)		(No ppt)	Maroon	Intense blue	Green.
Picratol	Yellow		Brown	Maroon		
Ednatol	None	Orange	(No ppt)	Maroon	Intense blue	Orange.
Amatol	None		Brown	Maroon	Dirty green	Green.
Ammonal	None		Brown	Maroon	Dirty green	Green.
Pentolite	None		(No ppt)	Maroon	Dirty green	Green.
Trimonite	Yellow	Red	(No ppt)	Orange	Red	
Tridite	Yellow	Red	(No ppt)	Orange		
Black powder[3]	None	No change			Blue	Green.

[1] Color appears immediately.
[2] Sometimes explodes mildly (puffs) upon addition of sulfuric acid.
[3] Tests of dried water extract.

place on a hard surface (iron, cement, stone, etc). Hit a light blow with a hammer. If it detonates, treat very carefully, as it is a primary or initiating expl. If no expln occurs, take a new sample and repeat with a hard blow. No deton means that it belongs to the same class of expls as TNT and only normal care is necessary. Another good test is to place one grain of a sample in a small flat mortar and rub gently with a pestle. If the sample detons, it belongs to primary or initiating expls

b) Place a small quantity of the expl on a non-combustible surface and insert the end of a safety fuse about 1 ft long. Ignite the end of the fuse and retire to a safe distance. If the expl deton, it should be considered as a primary expl and treated accordingly

5) *Solubility and Color Tests.* See the accompanying Table for data. Place 50 mg of the sample in a 5-ml beaker with 2–3ml of water and stir for 5 min before noting the color of the soln. Test the acidity with Universal pH indicator paper. Add a drop of Nessler's reagent (to 5g of KI in a min of cold water add a satd aq HgI_2 soln until a ppt forms; add 40 ml of 50% aq KOH, let settle, dilute to 100 ml with water, let settle and decant) and note color of ppt. Place 50 mg of sample in each of three depressions on a spot-test plate. To one add 2–3 drops of a 65–68% aq ethylene diamine soln, stir and note color of soln; to another add 3–4 drops of a solution of 1g diphenylamine in 100ml of concd sulfuric acid, stir and note color of soln after 1 min; to the last sample add 50mg of thymol and 3 drops of concd sulfuric acid, stir and note color after 5 min

6) *Melting point and range.* If the above characteristics are insufficient, it is advisable to determine approx the melting point, but not in the case of primary or initiating expls such as MF, LA, LSt, etc

Procedure:

a) Place a small quantity of the expl in a narrow test tube and attach a thermometer

by means of a rubber band in such a manner that the bottom of the tube and the mercury bulb are on the same level
b) Insert the lower part of the tube into a beaker containing light oil, heated by means of an alcohol lamp (or a Sterno outfit)
c) Observe the temp at which the expl liquefies
d) Cool the bath until the expl completely solidifies
e) Heat the bath to about 5° below the mp, as previously determined, and remove the flame
f) Resume heating with a very small flame, allowing the temp to rise about 1°C per min, until the expl just begins to melt
g) Remove the beaker from the flame quickly and observe the temp closely. If the melting was completed without raising the temp, the expl may be considered as pure (and not a mixture) and the temp can be considered as the mp
h) If the expl does not melt completely, place the beaker on a small flame and raise the temp 1° above that previously observed, if the expl still does not completely melt, raise another 1° and so on until the expl completely liquefies

If the temp between the beginning and the end of the melting is more than 3°, the expl can be considered to be a mixture, and this interval is called the **melting range**

All the tests described above should be conducted with very small quantities of expl which should not be confined in such a way that fragmentation of the container can occur
7) *Color Tests.* See "Color Reaction and Color Reagents", Vol **3**, p C405
Ref: Anon, "Military Explosives", **TM 9-1300-214/TO 11A-1-34**, Ch 12 (1967)

Fiery Mines. Same as Gaseous Mines

Figure of Insensitiveness Test. See FI Test

Figure of Merit is, accdg to Ref, gas volume times heat of explosion. If it is equal to 675 for TNT, for RDX it is 1162, Tritonal 792, Nitromethane 1133, Astrolite A-1-5 1600 & Astrolite G 1175
Ref: Bruce Anderson, Ordn **53**, 613–15 (1969)
Note: Astrolites are liquid military expls developed by the Explosives Corp of America, Issaquah, Washington. Their compns are based on Hydrazine

Filage (Étirage) des Poudres [French, literally meaning "Spinning (Drawing-out) of Propellants"]. This method used during manuf of French Smokeless Propellants ("poudres sans fumée", also known as "poudres colloidales") is described by Pepin Lehalleur (Ref 1). It is similar to *Graining*, described by Davis (Ref 2) and briefly in Vol **3** of Encycl, p C399

In the Fr method, also known as "moulage", the colloided NC, previously mixed in a kneader with other ingredients of proplnt, is forced, by means of hydraulic pressure of about 300 kg/sq cm, thru slots (of different widths and thickness for each type of proplnt), from which it emerges in the form of ribbons. They are then cut into strips of a length convenient for loading into the gun for which the proplnt is intended. These proplnts are known as "poudres lamellaires". For the formation of tubular proplnts (seldom used in France), the colloided mass is forced in the die into the space which surrounds a centrally fixed steel wire
Refs: 1) Pepin Lehalleur (1935), 298–300 2) Davis (1943), 302–04 3) P. Arribat et al, MP **32**, 279–85 (1950)

Filite. An Ital Ballistite, manufd in the form of cords. It contd equal parts of NG & Collodion Cotton with 0.5–1% aniline added. It was brown in color and had density 1.6. It was a "hot" proplnt which badly corroded gun barrels
Refs: 1) Gody (1907), 651 2) Marshall **1** (1917), 302 3) Encycl of Expls, PATR **2700**, Vol **2** (1962), p B9-L, under BALLISTITE

Filler. A material added to a product either to increase its bulk or weight, or to improve its props or appearance. In plastics, fillers are used to give the product certain desired

mechanical & electrical props, to improve internal stability, and to increase resistance to various service conditions. Some examples of fillers are as follows: wood flour, chopped paper (papier maché), palmetto, asbestos, chalk, cotton, mica, glass fibers, china clay, talc, graphite, metallic powders, metallic oxides & quartz
Ref: Kirk & Othmer **10**(1956), 799

Filler or Bursting Charge. The expl material which comprises the main chge in an expl loaded ordnance item
See also Charge in Vol **2**, p C150-L
Ref: NOLTR **1111**(1952), p G2

Filler or Bursting Charge of German Projectiles of WWII (Füllung or Füllpulver) (Fp or FP). See under German Military Explosives of WWII in this Vol

Filling of Bombs, Shells, etc. See under Loading of Ammunition

Film Bridge Initiators. Conductive films applied to surfaces of insulators by chemical precipitation or vacuum evaporation have been used to produce bridges which can be heated or exploded to initiate expl reactions. Since the initiation mechanism is complex, film bridges cannot be designed by computation. Typical complications include non-uniformity of film thickness and variations in conductive paths to the electrodes. Some experimental results have been obtained with a variety of film bridge initiators (See Ref 1), but the only type applied in standard fuzes has been the low energy graphite bridge type

Plugs for graphite bridge initiators are made by molding the plastic about a twisted pair of enameled wires, and then grinding the surface clean. This leaves a plastic surface with two metal islands separated by twice the thickness of the enamel. A droplet of colloidal suspension of graphite in water is applied over the point of closest approach of these islands to dry. Since both resistance and sensitivity are at least in part determined by the chance distribution of the graphite particles, item to item variation is great. Acceptable resistance range for Army items is 1000 to 10000 ohms, and for Navy 700 to 14000 ohms. Generally several hundred ergs of input energy are required for reliable initiation (Ref 2)
Refs: 1) W.M. Slie, "An Insensitive Substitute for Graphite Bridge Detonators for Laboratory Evaluation of Fuze Explosive Trains", NOL, NAVORD Report **4512** (Feb 1957) 2) Anon, Engineering Design Handbook, "Explosive Trains", **AMCP 706-179** (March 1969), pp 71-R to 72-L 3) Unidynamics Phoenix, Inc, PO Box 2990, Phoenix, Arizona 85062 (EED with thin film bridges) 4) G. Cohn , Edit, Expls&Pyrots **6**(7), 1973 (Abstract of Ref 3)

Filtration, Filter Aids and Filter Mediums.
Filtration is the separation of undissolved particulate solids from a mixture of fluid and solid. The separation is brought about by passage of the fluid thru a pervious septum *(filter medium)* in or on which the solids are retained. A driving force (gravity, vacuum, pressure, or centrifugal force) produces the flow. *Filter aids* may be added to the fluid before filtering to counterbalance the unfavorable characteristics of badly filtering materials

Filter Aids are porous powders added in small quantities to a soln or suspension to be filtered, in order to increase the efficiency of filtration. They are useful when handling suspensions of finely divided solids or of colloidal materials. The aid should not be much heavier than the liquids to be filtered, so that it will not settle immediately but will remain in suspension for a while. Kieselguhr and paper pulp are the most widely used filter aids. Others are sawdust, charcoal, Fuller's earth, magnesia, salt, gypsum and some activated charcoals. The charcoals act as decolorizers at the same time

Filter Mediums are materials used for filtering, such as filter paper, filter pulps, cotton fabrics, cotton ducks, cotton twills, cotton chain, cotton batting, wool cloths

and felts, jute cloths, fibrous glass, nitrated cotton cloths, human hair cloths, silk, nylon, vinyon, metal fabrics, porous blocks (such as carbon and graphite), aloxite, alundum, silica, porcelain and granular beds
Refs: 1) A. Weissberger, Edit, "Technique of Organic Chemistry", Vol **3**, 2nd edit (1956), pp 607–786 2) Perry (1963), pp 19-54 to 19-86 3) Kirk & Othmer, 2nd Edition, Vol **9**(1966), pp 264–86(23 refs)

FILUP (Franklin Institute Laboratories Universal Pulser). This instrument is used for testing Electro-Explosive Devices (EED's) (See in Vol **5** of Encycl, p E63-L). It supplies constant current or constant voltage pulses of varying magnitude & duration. It is also capable of measuring EED's resistance and functioning times and provides containment for the initiator being tested. The instrument is described in Ref 2 and a more recent version is described in Ref 3
Refs: 1) J.C. Kenyon, "No-Fire Level Test of TADM Electro-Explosive Devices", Ordnance Mission Data Rept (1962), White Sands Missile Range, New Mexico (DA Project 512–15–009) 2) C.T. Davey, "FILUP, Instructions for Operation and Maintenance", Franklin Institute Report **FM–A2357–1**, July 1961, prepared for Picatinny Arsenal under contract DA–36–034–501–ORD–3115RD (AD–315 471) 3) C.T. Davey, "Operation and Maintenance Manual for FILUP 3", Franklin Institute Report **FM–B2230**, June 1965, prepared for NASA/Langley Research Center under contract NAS 1–4062

Fineness. In order to determine if a sample of NC is sufficiently pulped, the following procedure is used:
1) Transfer a 10g sample of the dried NC(see Drying of NC) to a 250ml beaker, add 100–150ml water and mix by swirling the beaker
2) Transfer the mixture to a 250ml glass or rubber stoppered measuring cylinder, graduated in 2ml divisions over a length of about 10 inches to the 250ml mark. Rinse the beaker with several portions of water and transfer the washings to the same 250ml graduate
3) Fill the graduate to about the 240 mark, stopper and shake vigorously
4) Remove the stopper and rinse it with a stream of water from a wash bottle. Rinse the inner wall of the cylinder in the same manner until the volume in the cylinder is exactly 250ml
5) Replace the stopper and allow the cylinder to stand undisturbed for exactly one hour
6) Read the volume in ml occupied by the NC layer and consider this as the fineness value. A value of 90 ± 10 is usually considered sstisfactory
Ref: US Spec JAN-N-244(1945), p 11 (Now MIL-N-244A(2) (Oct 1965)
NOTE: See requirements and tests g) under Cellulose Nitrate Analytical Procedures in Vol **2**, p C122-L

Fineness of Grind (Degree of Dispersion). The method intended to measure the "fineness of grind" is described as Method 4411.1 (Sept 1, 1965) of Federal Test Method Standard No 141a. The apparatus consists of a device known as the Hegman Gage

See also Table 1 showing Comparison of US, Tyler, British and German Sieve Series in Vol **1** of Encycl, p A674

The French "Fineness Test for Nitrocelluloses" is described under the title "Épreuve de finesse des coton-poudres" in Vol **5** of Encycl, pp E108-R to E109-L

Finissage des Poudres. French for "Finishing of Propellants". The term means the last operations in the manufacture of propellants, to make them ready for packing or for loading in ammunition. The "finissage" operations include *lissage* (smoothing), *plombinage* (glazing or graphiting), *mélange* (blending) and sometimes *bottelage* (packing) (Refs 1, 2 & 4). The process is similar to that described by Davis (Ref 3)
Refs: 1) Vennin, Burlot & Lécorché (1932), 598–600 2) Pepin Lehalleur (1935), 306 3) Davis (1943), 305–06 4) J. Fauveau & R. Delpy, MP **31**, 161–66(1949)

Fin Stabilization and Spin Stabilization.

Fin Stabilization is defined in Glossary (Ref 2, p 115-R) as the method of stabilizing a projectile, as a rocket, bomb or missile, during flight by the aerodynamic use of protruding fins

Spin Stabilization is defined in Ref 2, p 270-R as the method of stabilizing a projectile during flight by causing it to rotate about its own longitudinal axis

Accdg to Maj Hobart (Ref 3), the ides of fin stabilization is much older than spin stabilization. This is because the earliest weapons were smooth-bore and they could use finned projs, while spin projectiles required rifled weapons which were not invented until about 1500. The earliest fin-stabilized projectile was an arrow and then fireworks projs, which could be called rockets. The earliest projs fired from smooth-bore barrels of small arms or cannons were like balls and they did not have any stabilizing attachments. However, accdg to the description given in Milimet Manuscript of 1326, now in Christchurch, Oxford, the earliest cannon, known as *pot de feu* (fire pot) was fin-stabilized (See also Vol 2 of Encycl, p C26-L)

Maj Hobart further states that after 400 years of oblivion, the finned projectile has been reintroduced in the US in the *flechette* (qv). In the early 1960's this round was found to offer very considerable advantages

The spinning of a projectile is produced by the engraving of the bullet envelope on the grooves of the rifling cut into the bore of the barrel. The rate of rotation depends on the steepness of the rifling spiral (usually expressed as one turn in x inches; it may be referred to in larger guns as one turn in x calibers, or the helix angle may be the reference parameter)

Maj Ohart (Ref 1, p 331) discussed stability and accuracy of fin-stabilized and spin-stabilized rockets. Accdg to him, spin-stabilized rockets are more accurate than the fin-stabilized ones. On pp 341 to 349 are described various fin- and spin-stabilized rockets

Accdg to Maj Hobart (Ref 3, p 315), for a long-range weapon the spun projectile is superior to the fin-stabilized such as the "flechette", while for ranges up to 400 meters, the target effect is much the same, but the accuracy of the flechette depends entirely on launch conditions

More discussion on this subject is given under "Flechette"

A good example of successful fin-stabilized rocket is the *Faustpatrone* described in this Vol

Refs: 1) Ohart (1946), 331 & 341 to 349
2) Glossary of Ordn (1959), 115-R & 270-R
3) Maj F.W.A. Hobart, Ordn **57**, 313–15, Jan-Feb 1973

FIRE [Feuer (Ger), Feu (Fr), Fuoco (Ital), Fuego (Span) and Ogon' (Russ)]

Merriam-Webster (1961) gives the following definition of fire as a noun: "The phenomenon of combustion as manifested in light, flame and heat and in heating, destroying and altering effects"

The term "fire" can also be used to mean: the discharge of firearms (as in heavy fire), to set on fire, to cause an explosion by lighting or ignition (fire a charge of explosive), to detonate a charge of Dynamite or other Blasting Explosive, to propel as from a gun (fire a projectile, fire a rocket), to feed the fire (fire a boiler), to take fire (damp BkPdr will not fire), to discharge artillery or firearms and many other uses, as for example in "fire" meaning discharge from employment

The use of fire has been considered one of the basic elements of civilization along with tool making (frequently requiring the use of fire) and speech. Fire is generally used to provide either heat or light. Langhans (Ref 4) discusses the cultural and historical aspects of fire as a religious symbol, some aspects of the production of fire, and some early alchemical and scientific aspects, as well as theories of combustion. Greenwood (Ref 3) discusses fire as related to its fuels. Refs 1 & 2 are concerned with the heating effects of fire. Fire is considered under four topics: "liberated fire" (heating of objects), specific heat, latent heat (of changes of state), and "combined fire" (heat of reaction). Ref 2 is a fictionalized treatment of some aspects and effects of fire and includes an attempt to prepare charcoal as an ingredient for a subsequent preparation of BkPdr

Refs: 1) M.A. Pictet, "An Essay on Fire", E. Jeffery, London (1791) 2) J. Abbot, "The Role of Philosophy, Pt 5, Fire", Hagan and Thompson, Philadelphia (1845) 3) E. Greenwood, "Prometheus, USA", Harper&Bro, NY (1929) 4) Langhans, "Feuer", (Eine Kulturhistorische Studie), Suppl to SS **32** (1937), 46pp

Fire Aboard USS Forrestal in 1967, during which the bombs started to explode 90 seconds after the fire began, thus killing the first wave of fire-fighters, brought to prominence the necessity of increasing the "cook-off" period in bombs to at least 5 minutes. This task was achieved at the US Naval Ordnance Laboratory, White Oak, Md, 20910 by the method briefly described in Expls&Pyrots **3**(3)(1970). See also Fire Resistant Coatings for Bombs in this Vol

Firearm. Any weapon from which a shot is fired (discharged) by proplnts, but the term is usually applied to small arms. The invention of firearms dates to about the 14th century
Historical. See under Bullets, Historical, Vol **2**, p B-324
Refs: 1) C.W. Robinson, "Thermodynamics of Firearms", McGraw-Hill, NY (1943) 2)C.E. Balleisen, "Principles of Firearms", Wiley, NY (1945) 3) T.C. Whelan, "Small Arms Design" and "Ballistics", Small-Arms Technical Publishing Co, Plantersville, SC (1945–46) 4) Anon, "Fundamentals of Small Arms, **TM 9–2205**(1952) 5) Collier's Encyclopedia, Vol **8**(1957), pp 57–64 6) R. Held, "The Age of Firearms, a Pictorial History", Harper, NY (1957) 7) J.E. Hicks, "US Military Firearms", Hicks & Son Pubs, LaCanada, Calif (1962)

Fire Balls. The origin & date of first use of fire balls are unknown. Davis (Ref 2) quotes from the book of Hanzalet Lorrain in 1630 on how fire balls are made:

> Take a pound of sulfur, three pounds of saltpeter, half a pound of gum arabic, four ounces of orpiment; grind all together, and mix well by hand, and moisten with brandy and make into a stiff paste into which you will mix a half pound of ground glass, or of crystal in small grains, not in powder, which you will pass thru a screen or sieve. Then, mixing well with the said paste, you will form balls of it, of whatever size you please and as round as you can make them, and then you will let them dry. If you wish to have green fire, it is necessary merely to add a little verdigris to the composition. This is a very beautiful fire and thoroughly tested, and it needs no other primer to fire it than the end of a lighted match, for, as soon as the fire touches it, it inflames forthwith. It is beautiful in saluting a prince or nobleman to have such agreeable hand fire balls before setting off any other fireworks

Reilly (Ref 1) describes a gray mixt made by mixing 7 parts of meal powder with 100 parts of sulfur, and is mostly used for the manuf of fire balls (used in war). These consist of twill bags, charged with compressed gray mixture, which is filled into them with the aid of spirit; they are fired by means of a composition fuse. Gray mixture mixed with antimony sulfide is used for torches.

According to Col Fisher (Ref 3) incendiary balls were built up around a core such as small iron shot. Customary procedure was first to dip the shot into a vat of liquid sulfur, then wrap the ball in oakum, redip, roll in fine powder, and wrap with wire. This process was repeated until the diameter of the ball corresponded to the caliber of the cannon. A variant of the spherical surface-burning incendiary was the elongated projectile made by kneading a warm incendiary mix over a crossed iron frame which extended to approx twice the length of the desired diam. Typical incendiary components were green pitch, fine & corned powd, oakum, tallow, and a small quantity of naphtha. A fuze of fine powd was inserted in the nose to ensure ignition

Adaptations of burning-type incendiaries to metal projectiles began to appear toward the end of the 15th century
Refs: 1) J. Reilly, "Explosives, Matches and Fireworks", VanNostrand, NY (1938), p 147

2) Davis (1943), pp 55–57 3) Col J.B. Fisher, "Incendiary Warfare", McGraw-Hill, NY (1946), p 112 4) J.F.C. Fuller "A Military History of the Western World", Funk & Wagnalls, NY, Vol 2(1955), p 49, Footnote 5) J.R. Partington, "A History of Greek Fire and Gunpowder", Heffer & Sons, Ltd, Cambridge, England (1960), 6) H. Ellern, "Modern Pyrotechnics", Chem-PublgCo, NY (1961), p 87 (Not found in 1968 edition)

Fire Bombs. See Vol 4, pp D944-L to D948-L, under BOMBS AND BOMB COMPONENTS

Fire Bombs, Fillings. See Vol 4, pp D941-L to D944-L and AddnlRef: J.E. Roberts "Rubber Latex Fuels for Fire Bombs", Armament Res & Dev Est, GrBrit Mem (MX) **7/59** (Oct 1959)

Fire Control. The basic problem of fire control is concerned with launching a projectile from a weapon station (that may be moving) at a target (that may also be moving) so as to score a hit on the target. An element of probability enters the situation because during its flight the projectile is under the influence of natural phenomena outside the launcher's control. The variable factor in fire control problems is the lead angle between the line of sight from weapon to target and the weapon line (direction in which the weapon is pointed). The lead is affected by target motion, forces acting on the target during flight, and jump of the weapon in firing

Refs: 1) W. Wrigley and J. Hovorka, "Encyclopedia of Fire Control'', MassInstTech, 2 vols, (1957) 2) W. Wrigley and J. Hovorka, "Fire Control Principles", (1959), McGraw-Hill (NY), p 2

Firedamp. [Grisou (Fr), Wetter (Ger), Grisu (Ital), Grisú (Span), Gremuchii gaz (Russ)]. It is a combustible gas found in coal mines and also the combustible mixture of the gas with air. Firedamp began as marsh gas when the vegetation in the primeval swamp was laid down and the formation continued thru the transformation to peat, lignite and coal. Coal seams still produce firedamp, the quantity increasing with the rank of the coal

An analysis of 60 samples of firedamp from Belgian coal seams (Ref 1) showed CH_4 97, N 1.6, CO_2 0.84, H_2 0.019, H_2+N_2 0.047, C_2H_2 0.53%. Such a gas can easily form expl mistures with air and, if a source of ignition is available (See Firedamp Explosions), a serious expln can result. A firedamp expln can also stir up coal dust and set off a far more serious coal dust expln (See Coal Mine Explosions and Fires, Vol **3**, p C360

Refs: 1) J. Venter & P.S. Stassen, BurMines-InfoCirc **7670**(1953) & CA **48**, 1003 (1954) 2) E. Lensel, RevIndMinérale **46**(2), 102–15 (1964) & CA **64**, 3254 (1966)

Firedamp Dynamite (Dynamite Safe Against Firedamp) or **Permissible Dynamite** [Permitted Dynamite (Brit), Dynamite-grisou (Fr), Wetterdynamit (Ger), Dinamite-grisu (Ital), Dinamita grisú (Span) and Predokhranitel'nyi Dinamit (Russ)]. See in Vol 3, pp C444-R to C456-R under "Coal Mining Explosives, Permissible" and in Vol 5, pp D1603-L to D1604-R, under DYNAMITE; also Vol 1, pp A355-L to A356-R, under AMMONIUM NITRATE DYNAMITE

Firedamp Explosions. Firedamp may be ignited by the flame from an explosive (Ref 2) or detonator (Ref 4), by jets of compressed air (Ref 5), and by friction (Ref 6). Ignition of firedamp by expls is avoided by use of especially formulated expls called Permissibles. These expls usually contain inorg salts that absorb heat and thus cool the deton gases. The cooled gases in turn help to choke the flame of the firedamp (Ref 8). The salt may also inhibit chain propagation in combustion of CH_4 (Ref 1). NaCl seems to be the most effective additive for inhibiting firedamp ignition (Refs 1, 3, 5, 7, 8, 9 & 10). As little as 3% has a measurable

but inconsistent effect while 10–20% has a very definite effect. For best results the salt should be as fine as possible (Ref 9) (See also Coal Mine Explosions and Fires, Vol 3, p C360; Coal Mine Explosions, Measures for Prevention, Vol 3, p C364; Coal Mines, Determination of Firedamp and Coal Dust in Atmosphere of, Vol 3, p C367; Coal Mine Explosives, Testing for Permissibility, Vol 3, p C368; and Commercial or Industrial Explosives, Vol 3, p C434)

Refs: 1) A.I. Gol'bendr, DokladyAkadNauk **59**, 261%3 (1948) & CA **43**, 3199 (1949) 2) R.L. Grant & C.M. Mason, BurMinesRept-Invest **5049** (1954) & CA **48**, 7902 (1954) 3) P. Speeckaert, Explosifs(Liège) **5**, No 2, 65–72 (1952) & CA **48**, 11059 (1954) 4) J.E. Dolan, JApplChem **52**, 15–23 (1955) & CA **49**, 15240 & 15241 (1955) 5) R. Loison & M. Giltaire, RevIndMinérale **36**, 21–32 (1955) & CA **49**, 9279 (1955) 6) H. Titman & A.H.A. Wynn, RevIndMinérale **36**, 50–61 (1955) & CA **49**, 9279 (1955) 7) R. Loison & J. Cocu, RefIndMinérale **39**, 467–76 (1957) & CA **51**, 18609 (1957) 8) W. Taylor, RevIndMinérale **39**, 467–76 (1957) & CA **51**, 1860 (1957) 9) N.E. Hanna, G.H. Damon & R.W. Van Dolah, BurMinesReptInvest **5683** (1960) & CA **55**, 3981 (1961) 10) A.P. Glazkova & V.B. Balolev, VzryvnoeDelo **1966**, (60/17) 5–20 & CA **67**, 13426 (1967)

Firedamp-proof Detonators. Firedamp-proof detonators have not received the attention that firedamp-proof expls have, possibly because the expln of the detonator is lost in the immediately succeeding expln of the main charge. Treatment of the detonator charge in caps follows similar lines to treatment of Dynamites in the addition of cooling additives, such as salts or wax (Ref 1), BuOAc (butyl acetate) (Ref 2), or polycarboxylic acids, oxygenated polycarboxylic acids, halogen substituted polycarboxylic and oxygenated polycarboxylic acids, and the neutral and acid salts of these (Ref 4)

Doubling the thickness of the Cu shell of a cap reduces the hazard of igniting firedamp and replacing the Cu with steel reduces the hazard even more (Ref 6). The hazard from an electric blasting cap depends on the nature, size and density of the cap charge. Hazard from short-delays is governed by the type of the delay charge and whether or not there is slag ejection

Detonating Cord type materials have the PETN filler mixed with carbonates or salts of polycarboxylic acids or their halide or OH derivatives (Ref 3) or are sheathed in a $NaHCO_3$-NaCl mixture (Ref 7)

Refs: 1) H. Elsner, GerP 803645 (1951) & CA **46**, 1260 (1952) 2) F. Habbel & H. Elsner, GerP 889575 (1954) & CA **52**, 11426 (1958) 3) F. Habbel & J. Prior, GerP 919094 (1954) & CA **52**, 11426 (1958) 4) J. Prior, GerP 919156 (1954) & CA **52**, 14172 (1958) 5) J.E. Dolan, JApplChem **5**, 215–23 (1955) & CA **49**, 15240 & 15241 (1955) 6) N.L. Rosinskii, N.D. Yalovaya & P.I. Perkin, Bezopasnost' Truda v Prom, **3** (10), 30–1 (1959) & CA **54**, 15933 (1960) 7) W. Helfgin & W. Rohde, GerP 1916685 (1970) & CA **74**, 55857 (1971)

Firedamp-proof Explosives. Same as Permissible Explosives, described under the same Refs as listed under Firedamp Dynamite

Firedamp Recovery. Firedamp is essentially the same as natural gas and has value as a fuel. Recovery is practiced in Europe both for safety purposes (firedamp removed from the mine is no longer an expln hazard in the mine) and as a by-product for economic reasons. Recovery by several methods is possible. Some of these are cross-measure borehole methods from working galleries, boreholes from roads outside the seam being worked, superjacent heading method, suction, pack cavity method and blowers (Ref 3)

Refs: 1) J.I. Graham, Colliery Guardian **159**, 582–5 (1939) & CA **34**, 3089 (1940) 2) L.T. Minchin, GasWorld **133**, 211–13 (1951) & CA **45**, 3580 (1951) 3) J. Venter & P. Stassen, BurMinesInfoCirc **7670** (1953) & CA **48**, 1003 (1954) 4) E. Lensel, RevInd-Minérale **46** (2), 103–15 (1964) & CA **64**, 3254 (1966)

Fire, Greek [Feu grégeois (Fr), Grecheskii Ogon' (Russ)]. It is described in Vol 2, p B166-L, under BLACK POWDER, but the spelling of inventor of fire was wrong. It should be, accdg to Andreev (Ref 2), Kallinnikos

Refs: 1) Daniel (1902), 299(Feu grégois) 2) K.K. Andreev, "Vzryv i Vzryvchatyiye Veshchestva" (Explosion and Explosive Substances), VoyenIzdatMinistOborony, Moscow (1956) (Grecheskii Ogon')

Fire Hazards, Common. See under "Fires and Explosions in Industries" in this Vol

Fire Hazards, Uncommon. These are typically taken to be gases, solvents, and other chemicals that can burn readily or that can react with some other material to produce heat and so set themselves on fire. A few examples of such materials are Na, K, phosphides, concd acids and alkalies & ferrosilicon etc

Refs: 1) Kirk & Othmer, Vol 6(1951), pp 531–43 and in 2nd Edit, Vol 9(1966), 288–96 2) Sax (1957), pp 129–45 and in 3rd Edit (1968), 208–26

Fire Protection, Industrial. See under "Fires and Explosions in Industries" in this Vol

Fire Resistance Tests. They include the tests described in Vol 1, pp XXII & XXIII, under "SENSITIVITY TO FLAME, HEAT, SPARKS, ELECTROSTATIC DISCHARGES, ETC." The following tests were developed at the USBurMines: a) Fuse Test b) Hemispherical Iron Dish Test and c) Red Hot Iron Test

Fire Resistant Coatings for Bombs. The length of time bombs can withstand the heat from a jet-fuel fire, before exploding, can be approx tripled. The technique is to paint them with an intumescent coating, .015 inch thick. When this coating is heated, it moves thru a phosphoric acid–carbon precursor stage to a decomposing ester, which tends to form water, carbon and nonflammable gases. Blowing agents swell a thick carbon foam which envelopes the object in a heat-resistant layer

Temperatures were measured inside the casings of 750-lb bombs. 60 gallons of jet engine (turbine) fuel were burned in shallow pans underneath each bomb. Explosion temp – about 400°F – was reached in approx 1¼ minutes inside the uncoated casing and slightly less than 4 mins in the coated casing. In fires aboard aircraft carriers, or in ammunition supply depots, the additional time before detonation could be of great value to firefighters. The extra minutes might be sufficient to pull the bombs out of the flames – or, if that is impossible, for nearby personnel to seek safety

Refs: 1) Anon, "Research Trends", Summer 1970, Cornell Aeronautical Laboratory Inc, PO Box 235, Buffalo, NY 14221 2) Expls&Pyrots 3(11) (1970)

Fire Resistant Textiles. Clothing in general and working clothes in particular should be as fireproof as possible in order to reduce the danger of their catching fire when approaching or coming in contact with open flame, sparks, dangerous chemicals, etc. Fabrics made from wool, silk and protein-like polymers are not very flammable, but all cellulosic materials (such as cotton, rayon, cellophane, etc) are

Attempts to fireproof cellulosic materials date from the 4th century BC, when vinegar was used as a fire-retardant for wood. From that time, nothing of importance was done until the beginning of the 19th century, when Gay-Lussac made some linen and hemp fabrics fire-resistant by dipping them in solutions of some salts, followed by drying. These salts were water-soluble and did not remain in fabrics after washing. Beginning about 1850, some retardants were introduced which remained in the fabric after washing (insoluble deposits). One of the most important improvements was made,

however, in the early 1930's when mixtures of halogenated organics and insoluble metal salts and oxides were introduced. These retardants found extensive use during WWII. The latest development, which appears to offer more promise for future use, is based on the chemical combination of the fire retardant with the cellulose molecules

Among water soluble retardants (nondurable type) may be cited: Amm bromide, Amm tungstate, Na vanadate, Li hydroxide, Na molybdate, K or Na hydroxide, Amm metavanadate, DiAmm phosphate & Zn chloride.

Among the insoluble deposits (durable type) are: ferric oxide, stannic oxide, Mn dioxide & Pb monoxide

Among chlorinated organo-metal oxide type may be cited the following mixture: 25 parts of "vehicle solids" (consisting of chlorinated paraffins 60%, plasticizer 20% & film-forming resin 20%) are stirred in a ball- or roller-mill with 25 parts of "dry solids" (consisting of Sb_2S_2 35, $CaCO_3$ 20, color pigments 30, ZnO 5 & mildewcide 10) and the resulting mixture triturated with 50 parts of petroleum solvents. The fabric is dipped in this mixture and then dried

In the fourth type of fire retardants, a chemical bond between the molecules of the fire retardant and cellulose should produce a finish that strongly resists the effects of laundering and weathering. Among such retardants may be cited cellulose-ammonium phosphate, cellulose-urea phosphate, cellulose-titanium complexes & cellulose-titanium-antimony finishes

A very good description of fire-retardant textiles is given in Kirk & Othmer (Ref 7)

Tests for Fire-resistance and Flammability. In the "fire-resistance test", also called "vertical burning test", a 12 x 2½ inch strip of fabric is suspended vertically so that the lower edge hangs just ¾ inch above the top of a gas burner (which has previously been adjusted to give a 1½ inch luminous flame). This means that ¾ inch of the fabric extends into the flame. After 12 seconds the flame is removed and, if the fabric continues to glow, the duration in seconds is noted, as well as the length of the charred area. Most specifications allow an after flaming max of 2 seconds and an average char length (10 strips) of 3½ inches, with a max of 4½ inches for any strip (Refs 7 & 8)

Flammability test, known as the AATCC or "inclined flammability test", was developed in the USA during WWII and is as follows:

A 6 x 2 inch strip of fabric, clamped in a rack inclined at 45°, is exposed at its lowest part to the flame of a microjet burner for a period of 1 second. If the sample catches fire, the time of flame travel over 5 inches of the sample is used as an indication of the relative flammability of the fabric

This method, as well as other methods of testing, are discussed in Refs 5 & 6

Refs: 1) H. Bennett, "The Chemical Formulary", Van Nostrand, NY, Vols I to IX (1933 to 1951) (Fireproofing and Flameproofing) 2) Textile Research Institute, "Fireproofing of Textiles", NY (1943) 3) J.E. Ramsbottom, "The Fireproofing of Fabrics", HMSO, London (1947) 4) R.W. Little, "Flameproofing Textile Fabrics" (ACS Monograph No 104), Reinhold, NY (1947) 5) G.S. Buck, Jr, AmerDyestuffReporter **38**, 78–84 (1949) 6) Collective, IEC **42**, 414–44 (1950), "Flame Retarding of Textiles" (Includes six articles and numerous refs) 7) Kirk & Othmer, Vol **6** (1951), 543–58 and 2nd Edit, Vol **9** (1966), 300–313 8) US Army Spec 6–345 (1945) and Spec MIL-D-10860 (1952) (Duck, Cotton, Fire, Water, etc Resistant)

Fire Retardant Paints. Fire retardant paints are based on chlorinated rubber and chlorinated plasticizers with added SbO. These reduce the rate of spread of flames. Addn of $NH_4H_2PO_4$, PE, or dicyandiamide produces an "intumescent" or swelling paint that forms a thick insulating layer over the surface to which it is applied when exposed to flames

Fire retardant paints do not control fires and are no substitute for an automatic sprinkler system. They are best used where the only hazard is exposed, combustible, interior finish materials or in isolated buildings where sprinklers will not be installed. The paint must be applied at the rate specified on the container; if spread thinner the proper

measure of protection will not be obtained
Refs: 1) ACS, "Fire Retardant Paints", Advances in Chemical Series **9**(1954) 2) Factory Mutual System, "Handbook of Industrial Loss Prevention", 2nd Edit, McGraw-Hill, NY (1967), p 5:14–5 3) C.R. Martins, Ed, "Technology of Paints, Varnishes, and Lacquers", Reinhold, NY (1968), 202–03

Fire Starter M1, Used in Pyrotechnics. See Vol **4**, p D762-L

Fires in Coal Mines. See under Coal Mine Explosions in Vol **3**, pp C360-R to C367-R

Fires and Explosions are described in pamphlet by H. Freytag entitled "Feuer und Explosionen", Arbeitsministerium Nordrhein-Westfalen, Zentralinstitut für Arbeitsschutz, Druck W. Bertelsmann, W. Bielefeld (1949)

Fires and Explosions of Ammonium Nitrate, Including FGAN (Fertilizer Grade Ammonium Nitrate). See Vol **1** of Encycl, pp A357-L to A363-R

Fires and Explosions in Industries (Prevention, Protection and Extinction). In order to have fire, there must be present a combustible material (such as wood, coal, fuel oil, etc), an oxidizing agent (such as oxygen, nitrates, chlorates, perchlorates, peroxides etc), and sufficient heat to start the fire. If buildings are constructed of wood or cardboard, they provide enough combustible material to start a fire

Fires may be started by one or several of the following so-called "common fire hazards": matches and cigarets, rubbish (spontaneous ignition), locomotive sparks, mechanical spartks, friction, open-flame devices, heating appliances, electric wiring and equipment, static electricity and lightning. The primary object of fire prevention and extinction is to avoid loss of life and personal injury; the secondary objective is the protection of property

These problems are of particular importance in explosive, ammunition and propellant plants because a fire in these plants might be accompanied by an explosion with consequent loss of life and property
Refs: 1) R. Assheton, "History of Explosions", Institute of Makers of Explosives, NY (1930) 2) F. Shepperd, "Fire Chief's Handbook", Case-Mann-Shepperd Pubg Co, NY (1932) 3) Interstate Commerce Commission Regulations, "Transportation of Explosives and Other Dangerous Articles by Freight," NY (1941) and Supplements 4) A. Pordage, Ed, "Chemical Fires and Chemicals at Fires", Inst of Fire Engineers, London (1943) 5) R.C. Smart, "The Technology of Industrial Fire and Explosion Hazards", Chapman Hall, London (1947) 6) A.M. Cameron, "Chemistry in Relation to Fire Risk and Fire Extinction", Pitman, NY (1948) 7) Assoc Factory Fire Insurance Cos, "Properties of Flammable Liquids, Gases & Solids", Boston (1950) 8) Perry (1950), 1847–84, "Safety in Fire Protection", and in New Edition (1963), pp 24-88 to 24-94 9) Kirk & Othmer, Vol **6** (1951), 531–43, "Fire Prevention and Extinction" and in 2nd Edit, Vol **9**(1966), 286–99 10) Sax (1957), pp 129–45 and in New Edition (1968), pp 196–207 11) Pamphlets of Factory Insurance Association, Manufacturing Chemists Association, National Board of Fire Underwriters, National Fire Protection Association, National Safety Council, Underwriters Laboratories, Institute of Makers of Explosives, US Bureau of Mines, US Bureau of Standards, US Public Health Service, etc

Fires, Extinguishing (by Explosions). The extinguishing of fires by explosions is based on the mechanical action of the formed gases and on the blowing out of the flame. There is some application in oil and gas well fires
Ref: Yu. V. Mekker, KhimReferatZhur **1939**, No 11, pp 130–31 & CA **34**, 8284(1940)

Fires – Radioactivity and the Detection of Fires. An electronic detector apparatus responds to changes in current flowing thru an ionization cell containing an α-particle source. The presence of smoke particles, fumes, or vapor in the cell will change the current flowing in the apparatus and activate an alarm
Ref: M.E. Nahmias, MAF **18**, 613 (1954)

Fires and Spontaneous Ignitions. Spontaneous ignition of combustible materials results from some substance reacting with light, air, water or other chemical to produce heat. If the heat is not readily dissipated, the temp rises in the substance and ignition is likely. Cotton rag containing linseed oil is the most common example. Fresh hardwood charcoal, hay, grain, feed and manure, wet sawdust, and finely divided metals including Fe, Co, Ni and U are subject to spontaneous heating
Refs: 1) National Fire Protection Association, Standard No 492, "Materials Subject to Spontaneous Heating (Issued Periodically) 2) Kirk & Othmer, Vol **6** (1951), pp 531ff and 2nd Edition **9** (1966), p 293 3) NFPA, "Fire Protection Handbook", Boston, Mass, 13th edit (1971)

Fireworks. [Feuerwerk (Ger), Feu d'artifice (Fr), Fuoco artifiziale or Fuochi d'artifizio (Ital), Fuegos artificiales (Sp), Feywerk (Russ)].

Fireworks are displays of colored lights, noise and smoke used on occasions of public celebration; also the devices used for such displays

Fireworks are a part of the field of civilian pyrotechnics (Greek *pyr*, fire; *techne*, art); the rest of the field is railway fusees and torpedoes. Military pyrotechnics includes lights for signals and illumination, noise makers for training, heat sources, smokes, and some minor rocket effects. Many of the same formulas and devices are used in both fields with the military devices more sturdily built

The essential characteristic of these devices is that the mixtures used in them produce reactions that are exothermic, self-contained, and self-sustaining. Most fireworks are intended for immediate consumption and have little or no shelf life. The sale of fireworks to individuals not licensed to put on displays is forbidden in many parts of the USA for reasons of safety. See also Pyrotechnics
Refs: 1) A.D. & P. Vergnaud, "Nouveau Manuel Complet de l'Artificier Pyrotechnie Civile", Edit G. Petit, Paris (1906) (Manuels Boret) 2) A.St.H. Brock, "Pyrotechnics: The History and Art of Fireworks Making", Harrap Ltd, London (1922) 3) J. Pepin Lehalleur, "Poudres, Explosifs et Artifices", Baillière, Paris (1935), pp 465–78 4) G. Weingart, "Dictionary and Manual of Fireworks", Weingart, New Orleans (1937) 5) J. Reilly, "Explosives, Matches and Fireworks", Van Nostrand, NY (1938) 6) T.L. Davis, "Pyrotechnic Snakes", JChemEduc **17**, 268–70 (1940) 7) A. Lotz, "Das Feuerwerk", Hiersemann, Leipzig (1940) 8) Davis (1943), 52–124 (Pyrotechnics) 9) G. Weingart, "Pyrotechnics, Civil and Military", ChemPubgCo, Brooklyn, NY (1943) and 2nd Editn (1947) 10) A.St.H. Brock, "A History of Fireworks", Harrop, London (1949) 11) A. Izzo, "Pirotecnia e Fuochi Artificiali", Hoepli, Milano (1950) 12) Kirk & Othmer, Vol **11** (1953), pp 322–32, "Military Pyrotechnics" and pp 332–38, "Commercial Pyrotechnics" 13) A.St.H. Brock, "The Art and Craft of Firework Making", Chem&Ind **1953**, 250–56 14) A.A. Shidlovskii, "Osnovy Pirotekhniki" (Foundations of Pyrotechnics), GosIzdatOboronProm, Moscow (1954). Engl translation as "Foundation of Pyrotechnics", AD 602687 from WPAFB, Ohio (1964) 15) Ellem, "Pyrotechnics", 1st Editn (1961) 16) Shidlovskii, "Foundations of Pyrotechnics", New Editn, Moscow (1964) 17) Ellem, Pyrotechnics, 2nd Edit (1968) 18) Kirk & Othmer, 2nd Editn, Vol **16** (1968), 824–40, "Pyrotechnics" 18) Rev R. Lancaster, T. Shimizu, R.E.A. Butler & R.G. Hall, "Fireworks, Principles and Practices", ChemPubgCo, NY (1972) 19) K.O. Brauer, "Handbook of Pyrotechnics", ChemPubgCo, NY (1973)

Fireworks Art Displays of Barry Rothman, who lives near Downingtown, Pennsylvania, are discussed by Al Haas in Philadelphia Inquirer, June 24, 1973, p 1gB and reprinted in Expls&Pyrots **6**(9), 1973

Firing of Blasting Explosives. Blasting Explosives fall into two classes: low or deflagrating and high or detonating. Blasting Explosives are described in Ref 1, pp B202 to B211

Low Explosives (such as Black Powder, described in Ref 1, pp B165-R to B177-R) are initiated by Fuses, Squibs and some Blasting Caps. High Explosives are initiated by Electric Blasting Caps, Detonating Cords (such as "Primacord" Bickford) and by Detonators

Refs: 1) Encycl of Expls, PATR **2700**, Vol **2** (1962), B185-L to B201-R (Blasting Caps); B212-R to B214-R (Blasting Squibs, Electirc) 2) Ditto, Vol **3**(1966), C229-R to C531-R (Cord Detonating) 3) Blasters' Hdb (1966), 87–95 [Electric Initiating Devices, which include: DuPont Electric Blasting Caps (EB Caps), Copper Wire Instantaneous, Iron Wire Instantaneous, Aluminum Wire Instantaneous, "SSS" Seismograph Caps, Delay EB Caps, "Acudet" Mark V Delays, MS Delays, Coal Mine Delays and Electric Squibs] 4) Ditto, 95–110 [Nonelectric Initiating Devices, which include: Blasting Caps, Safety Fuse, "Ignitacord", "Quarrycord", High Energy Detonating Fuse (such as "Primacord" Bickford, "E-Cord", Low Energy Detonating Cord, "LEDCORE" MS Delay Assemblies, and "LEDCORE" Seismic Delay Units)] 5) Encycl of Expls, Vol **4**(1969), D733-L to D742-L (Detonators, Igniters, and Primers Used for Nonmilitary Purposes)

Firing Composition 121. One of the mixts used by the Germans during WWII in igniters: Pb chromate 50, K chlorate 25 & silicon 25%
Refs: 1) PB Rept **95613**(1947), Section U 2) PATR **2510**(1958), p Ger 49-L

Firing Current Required to Ignite or Detonate an Initiating Device. When an electric current is passed thru a resistance wire (bridge) of an Electric Cap or an Electric Detonator, the heat developed in wire ignites the "ignition composition" (qv) or detonates the mixture contd in detonators. The current required to achieve this is known as the *firing current.* The easier the compn to initiate, the smaller is the current required for such purpose

There are also electric initiating devices, known as "bridgeless type electric cap". Such a cap, manufd by the Germans during WWII, is described in Vol **5**, p E55-L, including its cut. It contains a primer mixt of LA & LDNResorcinate, a pole piece, insulating cap, Pb/Sn foil washer (attached by shellac to an insulating material washer) and a contact ring. The cap required 120 to 160 volts to fire it.

NOLTR 1111 (1952), p 3,50 describes a British innovation, under development in the USA since WWII, which uses as a bridge a conductive carbon film rather than a wire in a primer. The carbon bridge primer offers several advantages over the bridge wire. Its high resistance makes it particularly suitable for circuits using a thyratron for triggering, and secondly is its ease of formation, eliminating the handling of fine wires. The major disadvantage is its sensitivity to electrostatic discharge which makes it hazardous to handle

In Table E5, p E28 of Vol **5** are given electrical characteristics of US Electric Bridge Type Delay Detonators XM60, T65 and T68 and compns of their spot, primary and secondary (base) charges

Other bridge-type electric initiating devices listed in Encycl include: Electric Blasting Initiators (Vol **2**, p B188); Electric Blasting Squibs (Vol **2**, p B213); Electric Detonators, Military (Vol **4**, pp D803 to D807), D846 to D850 and in Vol **5**, p E29-L; Electric Delay Primers (Vol **4**, pp D868 & D869); Electric Igniters (Vol **4**, p D733 & D734); Electric Igniter-Primers (Vol **4**, pp D1066 & D1067); Electric Fuses (Vol **5**, p E39-R); Electricity in Blasting (Vol **5**, pp E34 & E35); Electric

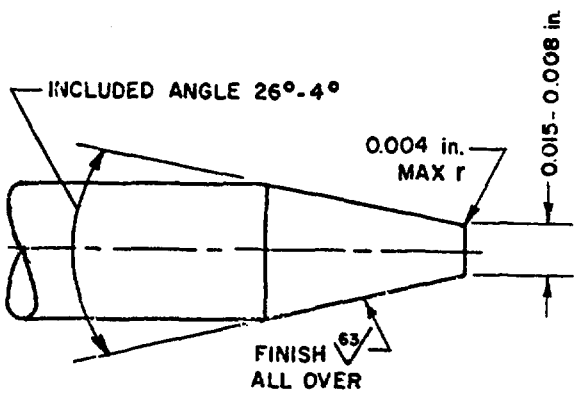


FIG F3 *Standard Firing Pin for Stab Initiators*

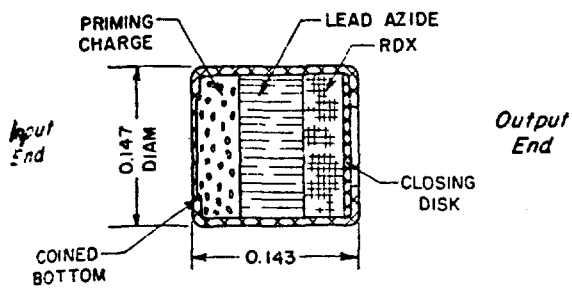


FIG F4 *Stab Detonator, M55*

Primers, Military (US) (Table E9 on p E57 gives electrical characteristics and compns of explosive trains of Electric Primers XM85, 87, 88 and 89); Electric Squib, MK1, All-Purposes (Vol 5, p E59-L); Electro-Explosive Devices (Vol 5, pp E63-L to E64-L)l Electro-Explosive Devices, Pulse Firing (Vol 5, p E65); Electro-Explosive Device, Thin Film for (Vol 5, p E68-L)

S. Odierno, "Information Pertaining to Fuzes", Vol 4(1964), Explosive Components has compiled a Table, "Electrical Detonator Data Sheets", showing electrical characteristics & firing times for various electrical detonators

See also Vol 5, p E38-L ff, "Electrostatic Discharges and Sensitivity of Explosives to Initiation by Them"

Firing Devices. See Vol 4, Section 3, Part C, pp D769-L to D773-L and Figs 22, 23, 24, 25, 26, 27 & 28

Firing Pin. In a mechanical fuze, contact sensing is converted directly into mechanical movement of a firing pin which in turn is driven into or against the first element of an expl train (primer). For the simplest solution to obtain initiation using this method, the forces on munition impact are used to crush its nose, thereby forcing the pin into the primer. In a base fuze, the pin may float in a guide thru which it moves during relative changes of momentum. The two types of initiation using these principles are *stab* and *percussion*

If the pin punctures the primer case and enters a suitable expl charge, an expln can occur. This is known as stab initiation. The shape of the firing pin commonly used in the US is constructed as shown in Fig F3 (Ref 1). Common firing pin materials include both steel and aluminum alloys. Rear end of pin may be shaped in any way convenient for assembly. Alignment of pin with respect to primer and surface finish of pin are important and affect the sensitivity (Ref 2). Fig F4 shows a typical stab detonator. For more information on stab detonators, see Vol 4, pp D844-L to D846-L

If the firing pin does not puncture the case, the initiation is called percussion. The difference in action is due to primer construction. In a percussion primer, the expl is backed up by a metal anvil (See Fig F5). For information about percussion detonators, see Vol 4, pp D852-L to D854-L. The firing pin dents the case and pinches the expl between case and anvil. A percussion firing pin that has a hemispherical tip (See Fig F6) gives greater sensitivity than a flat tip, but changing tip radius has little effect on primer sensitivity. For more information on sensitivity relationships with respect to cup, anvil, charge, and pin, see Ref 1. The effect of firing pin alignment on primer sensitivity indicates little effect if eccentricity is less than 0.02 inches. Above

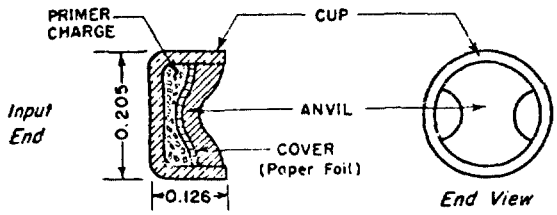


FIG F5 *Percussion Primer, M29A1*

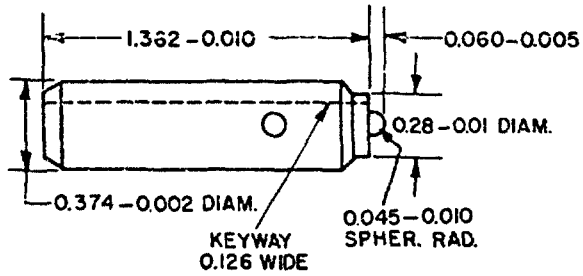


FIG F6 *Percussion Pin for Bomb Fuze, M904, to Initiate M9 Delay Element*

this figure, sensitivity decreases rapidly
Refs: 1) Anon, "Engineering Design Handbook, Explosive Trains", **AMCP 706-179** (March 1965) 2) Anon, "Engineering Design Handbook, "Fuzes, General and Mechanical", **AMCP 706-210** (November 1969)

Firing Range. An area or site designated for firing practice. Also the distance from a target at which a gun can be fired with effective results, as in "he was within firing range"; this range is considered as a capability of the gun
Ref: Glossary of Ordn (1959), 117-R

Firing Station, Guided Missile. A self-contained electronic item which provides remote control of guided missile functions prior to and including firing. It may include an "emergency propellant shutoff system". This is a self-contained unit designed to instantaneously disable the propulsion system of a guided missile
Ref: Glossary of Ordn (1959), 117-R & 226-L

Firing Systems. A group of connected components arranged to permit detonation of a chge from a remote station. The system may be electric, nonelectric, or a combination of both. In air, the system may be a simple fuse that leads from a distant point to a blasting cap, and a match with which to light the fuse. Underwater the system may be a complex arrangement of electrical wiring, blasting caps, detonating cord, primacord primers and a blasting machine. In either case, the firing system transmits energy from the point of firing to the main chge to detonate it

For underwater work, a combination system is recommended. Best results are obtd when the detonating cord is led from the main chge to a point above the surface of the water. An electric blasting cap is then attached to the cord and connected to the blasting machine or other power supply. This combination gives positive results because all electrical connections are above water
Ref: Anon, "Use of Explosives in Underwater Salvage", US Navy Dept, Bureau of Ordnance Pamphlet **DP 2081** (1956), Chapter 5, pp 28–38

Firing Time. A period of time required to fire a charge of explosive, propellant or pyrotechnic composition at various temperatures of heating or at various electric currents. The term is also applied to the period of time during which a weapon is fired (Ref 1)

In order to determine the "firing time" of an expl, proplnt or pyro vs temp of heating, a few grains of sample are placed on the surface of preheated, and maintained at desired constant temperature, devices used for determination of melting point. One of such devices is Fisher-Johns Apparatus [described in Fisher Scientific Co, Pittsburgh, Pa, catalog (1970), p 640]; another is Maquenne Block (electrically heated metal block with holes for capillary tubes & thermometer)

Several other methods are listed by Reilly (Ref 1) for detn of time of deflagration or expln. Among them the method of

E. Berl & G. Rueff, described in Cellulosechemie **14**, 43(1933). Their apparatus consisted of a copper block, heated electrically with holes for test tubes

The firing time test can also be conducted by immersing to a fixed depth in a bath of molten Wood's metal, (preheated to a desired temp and maintained constant) a metal Blasting Cap contg a small sample to test of const wt, say 0.02g. The time required to deflgr or explode is noted and the test repeated at various other temps of the bath (Ref 2, pp XVI & XVII). The lower is the temp of exposure the longer is the time required to deflgr or explode a sample. For example, if an unknown sample required 12 mins to be exploded at 168°, the time could be 16 mins at 160°, 30 mins at 148°, 60 mins at 143°, 2 hrs at 139.5° and 5 hrs at 136°C (Ref 1, p 83)

There are a few examples of "firing time vs electric current" listed in Vol **5**(Ref 4), Table E5, p E28 for Electric Delay Detonators XM60, T65 & T68 and in Table E9, p 57 for Electric Fuze Primers XM85, 87, 88 & 89

In Vol **2** of Encycl (Ref 3, pp B264-R & B265-L) is described "Bridge Wire Lag" and there is defined the term "Induction Period" (See also in this Vol "Firing Current Required to Ignite or Detonate an Initiating Device")

Refs: 1) Reilly (1938), 83 2) Encycl of Expls PATR **2700**, Vol **1** (1960) 3) Ditto, Vol **2** (1962) 4) Ditto, Vol **5**(1972)

First Aid is defined as the immediate, temporary treatment given in case of accident or sudden illness before the services of a nurse or physician can be secured

Every workplace should be provided with equipment and one or several persons who have received standard first aid training, for instance, from the American Red Cross or US Bur Mines

If a plant maintains a full-time physician and nurse service, then the organization of first aid crews is not of such great importance as in small plants not having hospitals and located at some distance from any. In the latter case, it is advisable to have a well trained first aid crew and equipment (See also under Industrial Hygiene and under Toxicology)

Refs: 1) US Bureau of Mines, "Manual of First Aid Instruction", Washington (1940) 2) W.M. Gafafer, "Manual of Industrial Hygiene", Saunders, Phila (1943) 3) American Red Cross, "First Aid Textbook", Blakiston, Phila (1945) 4) R. Clay, "A New System of First Aid", Faber, London (1946) 5) Fisher Scientific Co, "Laboratory First Aid and Fire Fighting", The Laboratory **16**, No 4, 90–95(1947) 6) S.C. Cullen & I.G. Gross, "Manual of Medical Emergencies", Year Book Publishers, Chicago, Ill (1949) 7) National Safety Council, "First Aid Service in Industry, Health Practice", Pamphlet No 8(1949) 8) A. Thorndike, "Manual of Bandaging, Strapping and Splinting", Lea & Febiger, Phila (1950) 9) J.H. Perry, Edit, "Chemical Engineers' Handbook", McGraw-Hill, NY (1950), pp 1875–1882 (Topic not included in 1963 edition) 10) R.E. Kirk & D.F. Othmer, Edits, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **6**(1951), pp 558–560: Anon, "First Aid" (36 references)(Topic not included in 1972 edition) 11) N.V. Steere, "Handbook of Laboratory Safety", The Chemical Rubber Co, Cleveland, Ohio (1967), pp 19–28, 61–68, 263 & 312

First-fire. In pyrotechnic devices, an easily ignitable compn between the fuse (or other igniter) and the main charge. The main charge is not easily ignited by the fuse alone. The same as Igniter Composition (See in Article on Pyrotechnics)

First Fire Compositions in Igniters. It can be a mixture of an illuminating compn with BkPdr. Five examples are listed in Vol **4**, p D759-R

First Fire, Ignition and Starter Mixtures Used in Pyrotechnics such as defined in Dr Ellern's book are listed in Vol **4**, pp D763 to D767 and in Tables F, G, H and I

First Fires, Igniters and Starters in Pyrotechnics. Their definitions are given by Dr H. Ellern in "Military and Civilian Pyrotechnics", ChemPubgCo, NY (1968), p 189. See Vol 4 of Encycl, p D760-L

Fischer, Karl (1894–). Ger chemist and a head of the firm manufg various chemicals and apparatus. Inventor of Karl Fischer Reagent for detn of moisture content (See Vol 5, pp D1622-L to D1628-R, under DYNAMITE
Ref: Staff, Explosvst 2, 46(1954)

Fischer-Tropsch, Ruhrchemie Synthesis is a catalytic process developed at Kaiser Wilhelm Institut in Germany before WWII (Ref 1) for the artificial production of mineral oil products (hydrocarbons). Essentially, the process consists of the following steps:
1) Water gas is obtained by the action of steam on glowing coal or coke

$$C + H_2O \rightarrow CO + H_2$$

2) Petroleum hydrocarbons are produced by heating $CO + H_2$ to about 200° at normal (or slightly increased) pressure in the presence of catalysts such as cobalt oxide etc. The following reactions take place:

$$nCO + 2nH_2 \rightarrow C_nH_{2n} + nH_2O \text{ and}$$
$$2nCO + nH_2O \rightarrow C_nH_{2n} + nCO_2$$

The product obtained by the Fischer-Tropsch process has been called ''Synthal''
Note: When zinc oxide catalyst is used, the resulting product is methanol
Refs: 1) F. Fischer, "Die Umwandlung der Kohle in Oele", Bortraeger, Berlin (1923) 1a) F. Fischer & H. Tropsch, Ber **56**, 2428 (1923); **59**, 830, 832 & 923(1926) 2) A.E. Dunston, Edit, "The Science of Petroleum", Oxford Univ Press, London (1938), **3**, p 2136 3) H.A. Shade et al, "The Fischer-Tropsch Process", PB Rept **363** (Inspection of the plant at Hames, near Lille, France) 4) National Research Council, "The Chemistry of Coal Utilization", J. Wiley, NY, **2**(1945), p 1797: H.H. Storch, "Synthesis of Hydrocarbons from Water Gas" 5) R.N. Shreve, "The Chemical Process Industries", McGraw-Hill, NY (1945), p 380 6) W.G. Frankenburg, V.I. Komarewsky & E.K. Rideal, "Advances in Catalysis", Academic Press, NY (1948), **1**, pp 115–156 7) R.N. Shreve, "Selected Process Industries", McGraw-Hill, NY (1950), 759 7a) P. Karrer, Organic Chemistry, Elsevier, Amsterdam (1950), p 43 8) Kirk & Othmer **6** (1951), 961, 965, 969 9) H.H. Storch, N. Golumbic & R.B. Anderson, "Fischer-Tropsch and Related Syntheses", J. Wiley, NY (1951) 10) CondChemDict (1961), 498-R

Fisher Melting Point Apparatus. See under Melting Point Determinations and in Catalog of Fisher Scientific Co, Pittsburgh, Pa (1970), p 640

Fission Bomb. See Vol 1, p A499-L under Atomic Bomb

Fission Reaction. See Vol 1, p A501-L, under Atomic (or Nuclear) Energy

Fitch & Reunert patented in 1884 BkPdr contg starch. It is listed in Vol 1, p A171-R as Amidon (Poudre à l')

FI Test (Figure of Insensitiveness Test)(Brit). An Impact Sensitiveness Test devised about 1908 by Dr G. Rotter at the British Government Research Establishment, Woolwich Arsenal. Important changes were introduced in the method in 1966 by Mortlock and Wilby (Ref 4). The original method and the modifications are described

The original FI expresses in terms of a standard expl the relative energies required to produce explns of equal degrees of completeness. A standard impact sensitivity apparatus is used with the impact area enclosed so that the volumes of gases generated in an expln can be measured as an indication of the degree of completeness of expln. Picric Acid (PA) is assigned a value of 100% which was the first "standard". Expls less sensitive than PA have FI's greater than 100:

TNT 120, DNT 175. Expls more sensitive than PA have values less than 100: MF 10, PETN 38 & RDX 64

In performing the test, a standard weight is dropped from a series of measured heights onto fixed volumes of the test and standard expls. Four samples are used at each height. The heights are increased until complete deton occurs. The percentage volumes of gas liberated at each height are plotted as ordinate against the heights as abscissa. The ratio of the areas under the curves for the test and standard expls is the FI (Refs 1 & 2)

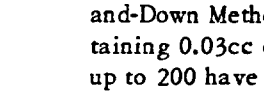
In 1966 several changes in the procedure were introduced by Mortlock and Wilby (Ref 4). Instead of measuring expln at several heights, the new procedure measures the height at which there is a 50% probability of expln. If there is no expln at a given height the next drop is at an increased height. If there is a shot at a given height, then the next drop is at a lower height. From these heights the median height is determined by the Bruceton Up-and-Down Method (See Vol 3, p C376 for info on Up-and-Down Method). Usually 50 shots containing 0.03cc of expl are used, although up to 200 have been shot. Standard and test expls are fired altemately. M & W also replaced PA with RDX as the standard expl. PA was an unsatisfactory standard because it showed day-to-day variations in behavior that seemed to be related to the humidity. RDX was assigned a value of 80 to give results consistent with previous work. In general the changes in procedure give an exact knowledge of the behavior of an expl at one impact distance while the older procedure gave less exact knowledge of behavior at several heights

The Rotter procedure and its modification, as described, give no information as to the nature of the reaction that is taking place in the expl, but show only that a certain amount of decomposition has occurred. An impact test is used as a guide to industrial handling practices and it is useful to know if gas is produced by a deton or a burn. A material that burns is likely to be safer than one that detonates. In 1954 Moore and Pape (Ref 3) pointed out this need and devised a remedy. They devised a slit and mirror arrangement under the anvil that permitted photography of the duration of light emitted by the decomposing expl. From this information slow burns, fast burns, and detons could be distinguished and handling hazards more accurately evaluated

Refs: 1) R. Robertson, JCS **119**, 1–29(1921) 2) J.L. Copp & A.R. Ubbelohde, TransRoy-Soc, **241A**, 248–65(1948) 3) P.W.J. Moore & R. Pape, "The Significance of the Figure of Insensitiveness obtained from Impact Tests on the Rotter Machine", ERDE Tech Memo No **14/M/54**(1954) 4) H.N. Mortlock & J. Wilby, Explosivst **14**(3), 49–55(1966)

Five-Foot Drop Test. One of the field tests to check the safety and operability of a fuze after mishandling. It is briefly described in Vol **4**, p D1093-R

FIVOLITE *(2,2,5,5-Tetramethylolcyclopentanol Pentanitrate or Nitropentanol)*



$CH_2.C(CH_2.ONO_2)_2$; mw 431.23, N 16.24%, OB to CO_2 −35.3%; crysts, mp 91–2°, sp gr 1.57. It was first prepd and patented in 1929 by Friederich & Flick (Ref 2). Accdg to Blatt (Ref 5), cyclopentanone is condensed with formaldehyde to form the ketone which is reduced with Cu chromite to secondary alcohol (pentanol) (mp 97.5°). This is followed by nitration with mixed nitric-sulfuric acid at 3° to Pentanitrate

Following are its props as listed in Refs 2, 3, 4, 5, 6 & 7:

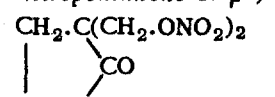
Detonation Velocity: 5060m/sec at d 0.75 & 7360 at d 1.57
FI (Figure of Insensitiveness) >TNT & <RDX
Heat of Combustion at C_v 1149.9kcal/mol
Heat of Explosion 1035kcal/kg
Heat of Formation +144.3kcal/mole
Impact Sensitivity with 2kg weight 26cm for 3/6 explns, vs 70cm for TNT
Power by Ballistic Mortar Test – 151% TNT
Power by Trauzl Lead Block Test 160% TNT

Stability at 100° by Abel Test – 8 mins

No info at our disposal about uses of Fivolite

Refs: 1) Beil, not found 2) W. Friederich & K. Flick, GerP 509118(1929) & CA **25**, 819(1931); USP 1962065(1934) & CA **28**, 4910(1934) 3) V.L. Wöhler & J.F. Roth, SS **29**, 332–33(1934) 4) Davis (1943), 285–86 5) Blatt, OSRD **2014**(1944) 6) ADL, Pure Expl Compds, Part **2**(1947), 224 7) Urbański **2**(1965), 201

FIVONITE (FV) *(2,2,5,5-Tetramethylolcyclopentanone Tetranitrate,* called also *Nitropentanone or ρ* [6])



$CH_2.C(CH_2.ONO_2)_2$
$CH_2.C(CH_2.ONO_2)_2$

mw 384.22, N 14.58%, OB to CO_2 –45.8%; wh crysts of d 1.611, cast d 1.590, both at 20°; d of liq 1.499 at ft p; mp 68–70° (Blatt), 74° (Davis). It was first prepd and patented in 1929 by Friederich & Flick (Ref 2). Accdg to Blatt (Ref 5), it is prepd by condensation of cyclopentanone with formaldehyde in Ca hydroxide soln at pH 9 to furnish tetramethylolcyclopentanone in 95–100% yield. This is followed by nitration with 98.5% nitric acid and the resulting crude product is stabilized by melting under water, adding Na carbonate to alkalinity, rinsing with water and drying. Yield 93–95%. The polymorphic form is obtd in the form of monoclinic prisms by crystallizing Fivonite from acetone-alcohol. The crysts are stable at RT

Following are expl and other props, as listed in Refs:

Brisance by Plate Denting Test, 112% TNT for pressed FV at d 1.54 (vs TNT at 1.55)

Deflagration Temperature, 265°

Detonation Velocity, 7292m/sec at d 1.57; 6815 at d 1.44

Explosion Temperature. Accdg to OSRD 1767 it does not explode below 360° when placed on Wood's metal surface, while accdg to OSRD 1986 it explodes at 285°

FI (Figure of nsensitiveness), 62% PA

Heat of Combustion, 1093.5kcal/mole

Heat of Explosion, 820kcal/kg

Heat of Formation, +166.3kcal/mole

Hygroscopicity at 25°C, gains 0.22% at 100% RH (Relative Humidity)

Impact Sensitivity by Bruceton No 3 Apparatus, 50% positive in brass cups with 5kg weight at 90cm drop, vs 50cm for RDX

Power by Ballistic Mortar Test, 127% TNT

Power by Trauzl Lead Block Test, 129% TNT

Rate of Detonation. See Detonation Velocity

Rifle Bullet Test. In 7 trials conducted in Canada with charges placed in steel pipe nipples – 5 passed, 1 detonated and 1 partially detonated

Stability (Thermal) at 100°, not acid and no expln in 300 mins

Stability at 135°, acid in 55–60 min and no expln in 300 mins

Storage (Surveillance), no apparent change in stability after 3 months storage at 50°

Thermal Stability. See Stability (Thermal)

Toxicity. In laboratory work no headaches or skin disorders were observed during 5 months

Vacuum Stability at 100°, 5–10cc gas evolved from 5g sample in 48 hrs

Velocity of Detonation. See Detonation Velocity

Uses: Being a HE and good plasticizer for NC, it is suitable for use in some expl and proplnt mixtures

Following mixtures are known:

FV/EDNA–50/50, called EDNAFIVE (described under Ethylenedinitramine in this Vol, p E238 ff

FV/PETN–50/50 – mixture of FIVONITE with PETN, known as PENTAFIVE (will be described under P's)

FV/RDX–50/50, known as CYCLOFIVE (See Vol **3** of Encycl, p C594-L

Refs: 1) Beil, not found 2) W. Friederich & K. Flick, GerP 509118(1929) & CA **25**, 819(1931); USP 1962065(1934) & CA **28**, 4910(1934) 3) L. Wöhler & J.F Roth, SS **29**, 332–33(1934) 4) Davis (1943), 285–86 5) Blatt, OSRD **2014**(1944) 6) E.H. Eyster & D.P. MacDougall, OSRD **5627**(1945) (Evaluation of FV as a HE) 7) ADL, PureExplCompds, Part **2**(1947), 243 8) A.M. Soldate & R.M. Noyes, AnalChem **19**, 442–44(1947) 9) Urbański **2**(1965), 201–02

Fixed and Semifixed Ammunition and Their Comparison with Separate-Loaded Ammunition are given in Vol 1, p A385-L, under AMMUNITIONS AND WEAPONS OR ARMS. Some illustrations are given in Vol 2, pp C74 & C75 under CARTRIDGE, AMMUNITION and in Vol 4, pp D777-R to D792-R

Fixed and Semifixed Ammunition, Rounds of. Round or Complete round of fixed artillery ammunition means that cartridge with propellant and loaded shell are all in one unit

In Vol 4, pp D777-R to D792-R the following fixed and semifixed rounds are described and Figs are given:

37mm HE-T, SD (High-Explosive-Tracer, Self-Destroying), Fixed Round (Fig 33, p D778)

40mm HEI-T, SD (High-Explosive-Incendiary-Tracer, Self-Destroying), Fixed Round, (Fig 34, p D778)

75mm HEP-T, M349 (High-Explosive-Plastic-Tracer), Fixed Round, is described in conf TM9-1300-203-1 (1967)

75mm AP-T, M338A1 (Armor-Piercing-Tracer) Fixed Round, (Fig 35, p D779)

75mm APC-T, M61A1 (Armor-Piercing-Cap), Fixed Round, (Fig 36, p D780)

76mm Canister, M363, Fixed Round (Fig 37a, p D781)

76mm HVAP-DS-T (Hypervelocity-Armor-Piercing, Discarding Sabot-Tracer), Fixed Round, (Fig 37b, p D782)

90mm HEP-T, T142 (High-Explosive, Plastic-Tracer) is described in conf TM9-1300-203-1 (1967)

90mm APC-T, M82 (Armor-Piercing-Cap-Tracer), Fixed Round (Fig 38, p D782)

90mm HEAT, M348A1 (High-Explosive-Antitank), Fixed Round (Fig 39, p D783)

90mm HVAP-T, M332A1 (Hyper-Velocity-Armor-Piercing-Tracer), Fixed Round (Fig 40, p D784)

105mm APDS-T, M392A2 (Armor-Piercing, Discarding Sabot-Tracer), Semifixed Round (Fig 41, p D784)

105mm HEAT-T, M456, Semi-fixed Round (Fig 42, p D785)

105mm HEP-T, M327, Semifixed Round is described in conf TM9-1300-203-1 (1967)

105mm HE, M1, Semifixed Round for Howitzer (Fig 43a, p D785)

105mm HE, RA, XM548 (High-Explosive, Rocket Action), Semifixed Round (Fig 43b, p D786)

105mm APERS-T, XM546 (Anti-personnel-Tracer), Semifixed Round (Fig 43c, p D787)

152mm HEAT-T-MP, XM409E3 (High-Explosive-Antitank-Multipurpose), Fixed Round for Gun Cannon, M81 (Fig 44a, p D788)

152mm TP-T, XM411E3 (Target Practice-Tracer) Fixed Round (Fig 44b, p D789)

57mm Canister Fixed Round T25E1 for Recoilless Rifles (Fig 45, p D790)

75mm HEAT-T Fixed Round for Recoilless Rifle (Fig 46, p D790)

90mm APERS Canister Fixed Round XM590 Series for Recoilless Rifles (Fig 47, p D791)

105mm HEAT Fixed Round, M341 for Recoilless Rifles (Fig 48a, p D791)

106mm HEAT Fixed Round, M344A for Recoilless Rifles (Fig 49, p D792)

Fixed Round of Artillery Ammunition, Explosive Train in. See Vol 4, p D839 and Fig 1–21b

Fixed and Semi-Fixed Ammunition: Testing and Surveillance. The following description is taken from the "Safety and Storage Manual for Explosives and Ammunition"

These types of ammunition are usually divided into lots varying from 5000 to 25000 rounds

Packing. Rounds of ammunition are usually packed in wooden boxes, with or without metal liners, or in wooden boxes with each round in an airtight metal or fiber container.

Handling and Shipping should be done according to regulations set forth by the ICC

Storage. Fixed and semi-fixed ammunition is usually stored in "Magazines for Ammunition" (qv) but is also allowed to be stored in other magazines, such as those designed for smokeless propInts, explosives, primers and fuzes. The boxes should be piled according to the Ordnance Dept Regulations and no loose rounds of ammunition should be stored. All magazines should be provided with good ventilation

Maintenance. This includes derusting, repainting, repairing packing boxes and repacking whenever necessary. The same instructions should be followed as given under "Smokeless Propellant, Maintenance"
Surveillance. This includes a visual examination of all boxes in which ammunition is packed, and a detailed examination of at least 5 rounds taken from two or more boxes to detect signs of rust, corrosion, exudation, projectiles loose in cartridge case etc. In addition to this, a representative sample of powder has to be taken from each lot, once a year, and subjected to the same tests as Smokeless Propellant: Methyl Violet, 65.5°, Surveillance and 134.5° Heat Tests, as described under these tests, and every three years a representative sample is taken from each lot for ballistic (firing) tests
Sampling.
1) Select one round of ammun ition at random from each of three boxes which are representative of the lot. Remove these rounds from the magazine and carefully withdraw the projectile, using a barricade in case of fixed ammunition
2) Weigh and record the amount of powder in each cartridge case, blend carefully and spread on a clean cloth for examination. If no signs of deterioration are noted, take a sufficient sample for all the stability tests and destroy the rest
3) If the sample shows signs of deterioration, as evidenced by the presence of grains with a yellow or orange discoloration or spots, treat it in the same manner as described under "Smokeless Propellant, Sampling"
Examination of Projectile consists in removing the booster (behind a suitable barricade and by means of a special device). If the booster cup is found to be corroded or rusty, or if the charge shows exudation, the shell should be salvaged or renovated. If the inside of the projectile is satisfactory, the nose of it should be closed with a suitable adaptor, or an iron or steel plug, and saved
Renovation and Salvage. Ammunition which has deteriorated to such a stage that replacement of the fuzes, boosters, shell charges or propelling charges is necessary, has to be subjected to either "*renovation*" (qv) or "*salvage*" (qv)
Destruction. Fixed or semi-fixed ammunition rarely deteriorates to such an extent that it must be destroyed rather than salvaged or renovated. It may be necessary, however, to destroy loaded shells, such as those which are fuzed and too dangerous to unload. In this case, instructions given under "Separate Loaded Shell, Destruction" should be followed

If the propelling charge becomes deteriorated, it has to be removed from the cartridge and burned as described under "Smokeless Propellant, Destruction"
Safety Precautions. Fixed and Semi-fixed ammunition should never be dropped, rolled or thrown, and as long as this is observed, there is little danger in handling it
Refs: 1) US Army Chief of Ordnance, "Safety and Storage Manual for Explosives and Ammunition", OO Form No **5994**, Washington, DC (1928), Sect **XVI**, pp 1–10 2) US Dept of Army, "Care, Handling, Preservation and Destruction of Ammunition", Tech Manual **TM9-1300-209** (Nov 1964) 3) US Dept of Army, "Artillery Ammunition", Tech Manual **TM9-300-203** (April 1967), Chap 6, pp 6-1 to 6-2 (Destruction of Ammo) 4) US Army Materiel Command, "Safety Manual", AMC Regulation **AMCR 385-100** (April 1970)

Fixed Particle Size and Shape Explosives were prepd for an investigation of the effect of particle size on detonation properties of compressed charges. Crystallization solvents and techniques were developed for close control of particle size for TNT, PETN, NGu and AmmPicrate
Ref: G.A. Cave, N.J. Krotinger, and J.D. McCaleb, IEC **41**, 1286–90 (1949) & CA **43**, 6415 (1949)

Fk. German abbr for Flak which means "Flieger Abwehr Kanone" (Antiaircraft Cannon)
Ref: Glossary of Ordn (1959), p 118-L & R

Flaking is the term for continuous solidification of molten material. Batch solidification is called *casting*. The types of apparatus as described in the ref are:

1) Table flaker – a flat metal sheet with turned up edges and jacketed on the under side for coolant flow. It is used for ½–1 inch thick slabs

2) Vibrator type – a tray in which the bottom and one end curves up gently to be level with the top edges of the sides. An attached vibrator breaks up the solid and shakes it over the curved end of the tray to a discharge conveyor. Cakes may be up to 3 inches thick

3) Belt type – a metal belt floated on water receives the molten material which breaks into flakes as the belt turns down over the return roller at the discharge end. The cake is usually from 1/8 to 5/8 inch thick. Thicker cake may require use of a top belt for addnl cooling

4) Rotating drum – single and duble drum models are made. In either case the internally cooled drums turn thru the molten material picking up a layer that is flaked as it is scraped off by a doctor knife (or knives) into a discharge conveyor. Double drums are counter-rotating and the knives and discharge conveyor are between the drums. Cake may be from 1/64 to 1/4 inch thick

5) Rotating shelf – a vertical series of rotating annular tables carried by a central column that supplies coolant to the under side of the tables. One of two side columns supplies molten material to the trays and the other column carries removing knives and conveyors to remove the solid

A brief description of a single-drum flaker is given, together with Fig 6, on p D1560-L of Vol 5 of Encycl and of a double-drum flaker, together with Fig 7, on the same page

Ref: Perry (1963), p 11:42-3

Flame. Accdg to the Encyclopedia Britannica (1964) 9, 413–16, *flame*, as a phenomenon of the physical world, is generally understood to be burning gas. Its prerequisite is the formation of an explosive mixture, eg firedamp and air. If such a mixture is ignited (by spark or small flame), combustion spreads from the ignition source to the adjacent layer of the mixture, each point of the layer serving as an ignition source for the next adjacent layer. Thus, a combustion wave (flame front, combustion zone) is formed which propagates thru the gas mixture leaving behind hot, burned gas. The mixture propagates a flame only above a min and below a max % of gas (lower and upper limits of flammability, respectively). For firedamp the limits are 4 & 15%, respectively

The wave travels at some definite velocity (burning vel) against the unburned mixture. Velocity depends on the composition of the mixture being zero at the limits and at a max at some intermediate composition (eg 9.5% for firedamp). There are three types of flame:

Diffusion Flame. When a slow stream of fuel gas flows from a tube into the atmosphere, air diffuses across the boundary of the stream and forms an envelope of expl mixture around a core of gas. The core decreases in height until it disappears at some distance above the tube. It thus assumes the shape of a cone. On ignition, a flame front spreads thru the mixture and stabilizes itself around the cone of fuel gas. The hydrocarbons in common fuel gases "crack" to form free C & H. The shell of carbon-bearing gas so formed gives such flames their luminosity

Turbulent Jet Flame. When a gas stream issues from an orifice above a certain critical velocity, it breaks up into a turbulent jet that entrains the surrounding air. The flame of such a jet consists of random patches of combustion and no cohesive combustion surface exists

Flames of Streams of Explosive Mixtures. From some point of ignition, a combustion wave spreads out until it forms a continuous surface over the whole cross-section of the stream. The flame front is shaped by relative values of burning velocity and gas velocity. If burning velocity exceeds gas velocity, the flame flashes back; if the burning velocity is less than gas velocity, the flame blows off into the atmosphere and becomes extinct. The flame establishes itself when the velocities are equal

Flame of Explosion. Nearly all explns are accompanied by a flame, varying in temperature, color, duration and size. In many cases, the appearance of flame is undesirable and it is necessary to suppress it completely or to diminish either its brightness or its size and temperature. For instance, when firing cannon it is desirable that the flame or flash not be so bright and intense as to be visible to the enemy. On the other hand, when shooting expls in fiery coal mines, the brightness of the flame is of no importance, but it is necessary to have the flame sufficiently cool and of short duration so as to eliminate the danger of it igniting the firedamp

The problem of the flame of coal mine expls was investigated by Aguillon (1880), Mallard & LeChatelier (1888), Bichel (1899), Wilkoszewski (1907), Will (1909), Dautriche (1910), Taffanel & Dautriche (1912), Taffanel & LeFloch (1913), French Investigating Committee (Commission des Substances Explosives), Naoúm, Kast, Selle, Berthmann, Beyling, Schultze-Rhondorf, Schmerber, etc

The following is the résumé of their conclusions:

1) The duration of the flame is as important as its size and temperature

2) There is a certain degree of delay in ignition, or a certain period of high temperature required to cause ignition of firedamp, and expls with short duration of flame (such as most of the brisant expls) would not ignite the firedamp even if the temperature of the flame is above the flash point of the firedamp. However, if the velocity of detonation of such expls is very high (say above 7000m/sec), there is danger that a sudden compression of the surrounding air, caused by the detonation, might raise the temperature above the flash point of the firedamp and cause an expln

3) The expls with long, durable flame (as Black Powder) almost always ignite the firedamp, even if the temperature is comparatively low

4) The larger the charge of an expl, the greater the length and duration of flame, and therefore it is important to establish a certain maximum charge *(charge limit)* for each expl used in fiery coal mines

5) The flames from permissible expls have shorter duration than those of other types and that is the main reason why they are safer to use; however, if the charge limit established for these expls is exceeded, there is danger of igniting the firedamp

6) The temperature of the flame is also of great importance and most countries have established a certain max temperature. Mallard and LeChatelier thought that the max should be 2200°, but the French Investigating Committee (Commission des Substances Explosives) lowered this limit to 1500° for "couche" expls (permissible in coal seams) and 1900° for "roche" expls (used for blasting accompanying rock). One of the best methods for lowering the temperature without reducing the efficiency of an expl is to incorporate in the expl a large proportion of AN (Ammonium Nitrate). Na or K nitrates or chlorides are also effective

7) The photographs of flames produced by mining expls, taken by Laffitte, showed that there are three classes of flames: a. Primary flames, due to reactions taking place within the borehole, b. Secondary flames, due to reactions in the gases outside the borehole but without the assistance of the air, c. Tertiary flames, due to the interaction of the oxygen of the air with the combustible gases from the expln. These flames often arise when the gases impinge on the walls of a shaft. It is only recently that the existence of secondary flames has been noticed. Previous investigwtors, such as Bichel, Will etc, believed that there were only primary and secondary flames; their "secondary" corresponding to what is now termed "tertiary"

The same phenomenon is observed on firing cannon and the flame which is produced on contact with the air is sometimes called *afterglow*. The "afterglow", which is undesirable because it might indicate to the enemy the position of artillery, may be partly or nearly completely suppressed by the addition of a few percent of alkali salt (or other flash reducing compound) to the propellant (See Flash Reducing Agents)

8) As the result of Laffitte's experiments,

Bichel came to the conclusion that, in order to judge the safety of an expl, it is important to know the ratio of the *time of detonation of the charge to the duration of the flame*, which he called *after-flame ratio* (Ref 1, pp 596–97)

Following are some of the results of Bichel's flame measurements using a 100g sample of expl:

Table F4

100g of Explosive	1 Time of Detonation (seconds)	2 Duration of Flame (seconds)	3 Length of Flame (mm)	4 After-flame Ratio (Item 1 divided by Item 2)
Black Powder	.0000233	.077	110	1:330
Blasting Gelatin	.000011	.0097	224	1:882
Guhr Dynamite	.0000134	.0083	228	1:620
Gelignite	.0000119	.0012	150	1:101
TNT	.00000119	.0016	108	1:1347
PA	.00000143	.0015	110	1:1049
Guncotton	.000016	.0013	97	1:81
Donarite	.0000267	.00040	69	1:15
Carbonite	.0000508	.00033	40	1:6.5

Note: The safer the expl from the point of view of firedamp ignition, the larger is the "after-flame ratio" and the lower duration of flame and its length (See Donarite and Carbonite)

9) Another factor which might contribute to the length, duration and temperature of the flame is the density of the expl. The higher the density, the higher is the velocity of detonation and temperature of the flame and the shorter is its length and duration. For this reason, permissible expls are made with very low density

10) Other things being equal, an expl with a high velocity of detonation may be expected to give a flame of less duration than a slow one. But a very high vel of deton is undesirable in a coal mining expl, because it is liable to ignite firedamp by the rise of temp caused by the sudden compression (Ref 1, p 596)

11) As there are many factors contributing to the flammability of firedamp by the flame of expls, most countries test the expls intended for use in fiery mines in special testing galleries in which the actual conditions existing in mines are simulated as nearly as possible (See Vol **3** of Encycl, pp C368 to C378-L)

Refs: 1) Marshall **2**(1917), 596–605 2) Naoúm, NG(1928), 384–85 3) Vennin, Burlot & Lécorché(1932), 228–35 4) Stettbacher (1933), 65–8 & 245–48 5) Pérez Ara(1945), 125–29 (Caracteras de la llama de la explosion) 6) H.G. Wolfhard & W.G. Parker, "Combustion Processes in Flames", Royal Aircraft Establishment Report, No **Chem 457**(March 1949) 7) R. Steinberger, IEC **48**, 766–68(1956)(Flames from nitrate esters; a review of recent work) 8) S.S. Penner & T.A. Jacobs, "Combustion and Flames", CalTech, Jet Propulsion Center, TechRept No **29**(1959) 9) M.G. Saunders & A.G. Smith, JApplPhys **24**, 115–17(1956)(Phase contrast observations in flames) 10) R.R. John & M. Summerfield, Jet Propulsion **27**, 169–175 & 178 (1957)(Effect of turbulence on flame radiation) 11) H. Selle, Explosivstoffe **8**, 9 & 195–204(1960)(Investigations on flame

phenomena in explosive conversions) 12) B. Lewis & G. von Elbe, "Combustion, Flames and Explosions in Gases", Academic Press, NY (2nd Editn 1961) 13) Ellern, Pyrotechnics, 2nd Ed (1968), 230–32 (High temperature flames) [See also "Burning Characteristics", in Vol **2** of Encycl, pp B343-L to B362-L and "Combustion" in Vol **3**, pp C425-L to C433-R]

Flame; Behavior Towards It of Explosives. A simple test to determine ignitability of expls by flame, proposed by Ficheroulle & Kovache, was as follows:

A sample (35g) of explosive (or propellant) is placed in the center of a sheet of combustible paper and one of its corners is ignited. Then observe the behavior of expl (or proplnt) when the flame reaches it. Some substances burn slowly, others rapidly, while primary or initiating expls detonate
Ref: H. Ficheroulle & A. Kovache, MP **32**, 135 (1950) [Compare with French "Epreuve de sensibilité à l'inflammation" ou "Aptitude à l'inflammation", known as Combustion en tas conique (Combustion in a Conical Pile) in Vol **5**, p E123-R. It is one of the French Official Tests] (See also "Burning Tests" in Vol **1**, p IX)

Flame Duration and Length. See Flame Length and Duration

Flame, Extinction of. Flames of any kind are very dangerous, especially if they come in contact with some explosive or highly combustible materials. When a flame comes in contact with an expl gas mixture with air (like firedamp in coal mines), the expln might be propagated a long distance unless some precautionary measures are taken

Sprinkling of water is not always effective, but the extinction improves if some inorganic salts (such as chlorides, carbonates, acetates or phosphates) are in water, as was proposed by Thomas & Hochwalt (Ref 1). Pease (Ref 2) proposed coating the walls in fiery coal mines with K salts. Some other investigators proposed quenching the flames using some nonexplosive dusts, such as stone dusts, dust of various inorganic salts of K (Ref 3). Dufraisse et al (Ref 4) proposed using K oxalate and claimed that 400mg per liter of air was sufficient to extinguish a flame. Still better results were claimed to be obtd with a mixt contg 30% K oxalate with 70% Na bicarbonate, because the amt required was only 200mg per liter of air. The value named by Dufraisse et al determined the "pouvoir" for 250 substances (Refs 4 & 5). Extinction of flames is also discussed by Lewis & von Elbe (Ref 6)
Refs: 1) C.A. Thomas & C.A. Hochwalt, IEC **20**, 575–77 (1928) 2) R.N. Pease, JACS **51**, 1839–56 (1929) 3) W.P. Jorissen et al, RecTravChim **52**, 403–12 (1933) 4) Ch. Dufraisse & M. German, CR **207**, 1221 (1938) 5) Ch. Dufraisse et al, CR **236**, 164 (1953) 6) B. Lewis & G. von Elbe, "Combustion, Flames and Explosion in Gases", 2nd Editn, Academic Press, NY (1961), 322–23 (Dust quenching occurs at a critical value of the surface area of the dust per unit vol of the suspension, and depends on the nature of the salt. Better results are obtained with salts having a mp under 200°. Alkali halides are better than carbonates, potassium better than sodium, fluoride better than iodide and better than chloride. If the dust concentration is high enough, even detonation waves can be extinguished)

Flame Length and Duration Measurements. In order to judge the approx length and duration of flame, Wilkoszewski, Will, Bichel, Kast, Naoúm and others (Refs 1–6) photographed, at the beginning of this century, various explosions, using ordinary cameras and films. Such photographs did not yield as useful data as was hoped. Although it was easy by such methods to distinguish between explns produced by coal mining expls and brisant expls, it was not possible to draw conclusions as to the relative safety of different coal mining expls. One reason for this was that the films available at that time were not sensitive to infra-red rays

emitted by comparatively cool flames which are, however, quite capable of igniting firedamp

More recent methods of photographing flames include the so-called "Schlieren-Method" and the "Motion-Picture Camera Method" using films sensitive to infra-red. These methods are described in Refs 7–9 and the definition of "Schlieren-Method" is given in Vol 2 of Encycl, pp C15-R & C16-L. The motion picture cameras are similar to those used in the movie industry (Ref 8)

In Vol 1, p XII are listed several flame tests (including 5 refs) and among them the Bureau of Mines Test, described in USBur-MinesBull **346**(1931), p 67

Belgrano (Ref 12) described the rotating drum apparatus (shown here as Fig F7) for measuring length (lungezza) and duration (durata) of flame (fiamma) produced on explosion. It consists of the following parts: electric motor (elettromotore) with speedometer (tachimetro), photographic apparatus (apparechio fotografico), electric transmission (transmissione elettrica) and mortar for firing (mortaio di sparo), located 6 meters from the apparatus. The mortar consists of a steel block with a hole for explosive charge

The photographic apparatus consists of camera protected from light (camera a tenuta di luce), which encloses a rotating drum with film (tamburo con pelicula), a screen with a slit (schermo con fessura), and a stand supporting the drum and transmission. At the front part camera, against the slit of the screen is located a lens (lente) which allows the passage of ultraviolet rays of the flame

The rotation of motor is regulated to cause the peripheral velocity of the drum to be 20 meters per second

Giorgio (Ref 13) lists rotating drum method and motion picture method and briefly describes the method of Berthmann & Käufer, without giving the information where the method was published, but just the year (1956). The Fig 32 given on p 99 of Ref 13 is shown here as Fig F8. It consists of: A = rotating drum apparatus with film, B = mortar made of marble, C = spherical vertical mirror and D = spherical horizontal mirror

US procedures for determining the characteristics of percussion primers include the rapid measurements of flame length, flame duration, flame temperature, etc in a special apparatus. It consists of two major units: the firing chamber (a closed box in which are mounted the sensing elements) and the electronic magnifying glass. It consists of the following parts:

A = rotating drum ensemble with a sensitive film
B = Mortar made of marble
C = Spherical vertical mirror and
D = Spherical horizontal mirror

The first of these mirrors placed near the borehole of mortar produces on the film

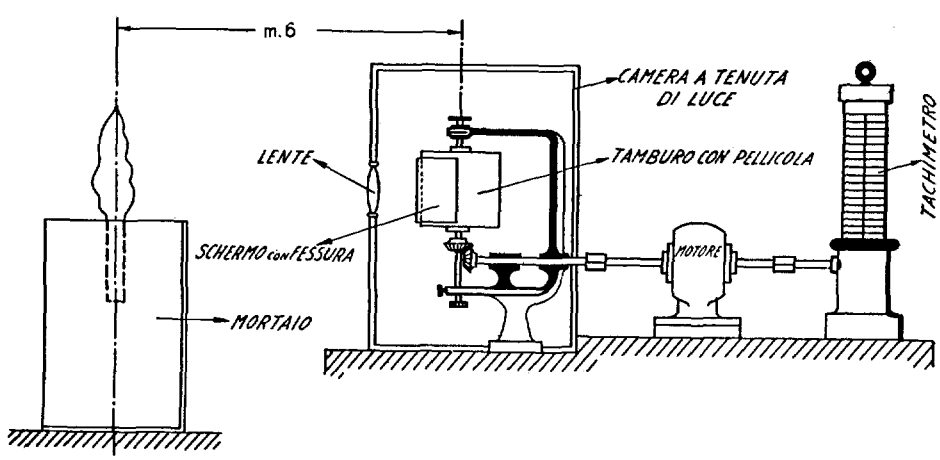
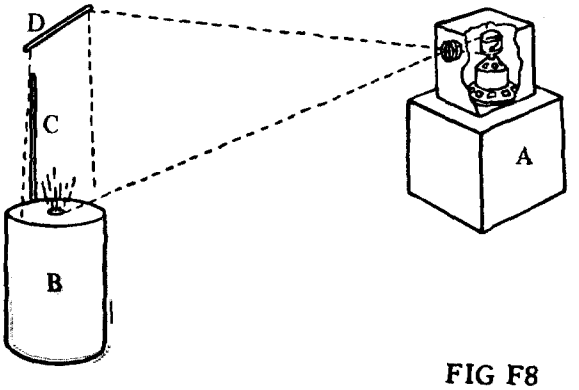
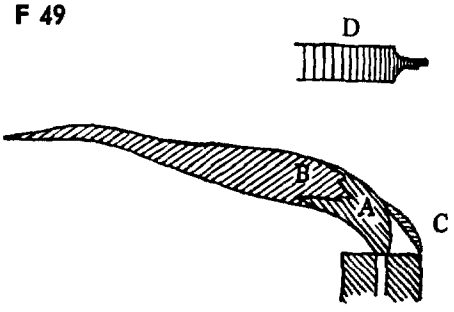


FIG F7

A = Rotating drum apparatus with film
B = Mortar made of marble
C = Spherical vertical mirror
D = Spherical horizontal mirror





A = Intense flame of expln
B = Successive luminosity
C = Image of vertical mirror
D = Image of horizontal mirror

FIG F8

of the drum notches permitting one to measure the flame; the 2nd of the mirrors reflects the course of the flame inside the borehole of the mortar even before it leaves the mouth. The results of work with this apparatus showed that the velocity, which is 2000–1000m/sec at the mouth, reaches 2500–2000m/sec outside the mortar

The drawing of flame shown in Fig 8 may be subdivided into: A = Intense flame of expln; B = Successive luminosity; C = Image of vertical mirror; and D = Image of horizontal mirror control panel. The parameters sensed by the apparatus are displayed on meters or lamps and are expressed as numbers which are arbitrary, but useful for comparing one primer with another. More information about this test is given in Refs 12a & 14

Refs: 1) C.E. Bichel, "Untersuchungsmethoden für Sprengstoffe", W. Erst, Berlin (1905). Its Engl translation, "Testing of Explosives", published in 1905 by A. Larnsen in London 2) Wilkoszewski, SS **2**, 141 (1907) & CA **2**, 899 (1908) 3) H. Kast & H. Selle, SS **3**, 23 & 153 (1908) 4) C.E. Bichel, SS **3**, 408–10 (1908) 5) W. Will, SS **4**, 323 & 343 (1909) 6) Marshall **2** (1917), 596–97 7) Ph. Naoúm & A. Berthmann, SS **25**, 193 (1930) 8) Marshall **3** (1932), 190–93 9) H. Schardin, "Das Toeplersche-Schlieren-Verfahren", Ver-DeutschIngenieurForschungsheft, July-Aug 1934, p 367 10) N.F. Barnes & S.L. Belinger, "Schlieren and Shadowgraph Equipment", JOpticalSocAmer **35** (8), Aug 1945 11) W.R. Keagy et al, "Schlieren Techniques for the Quantitative Study of Gas Mixing", Rand Corp, Santa Monica, Calif, **R-164** (1949) 11a) A.J. Zaehringer, RocketScience **4**, 65–6 (1950) (Flame photographs of Black Powder) 12) Camillo Belgrano, "Gli Esplosivi", U. Hoepli, Milano (1952), pp 171–72 (Apparatus for measuring length and duration of flame used by explosions) 12a) NOLTR **1111** (1952), 9–48 13) C. Giorgio, "Tecnica degli Esplosivi", DelBianco, Udine (1964), pp 98–100 (Study of flame of explosion) 14) Anon, "Explosives Trains", US Army Materiel Command Pamphlet **AMCP 706–179** (1965), pp 73 & 136

Flameless Cartridge Case of Cocking, mentioned in Vol **3**, p C388-R, 4th line from top consisted of a narrow fiber, wool, etc, cylindrical bag (impregnated with Amm chloride soln), in which was placed an explosive cartridge before loading into a borehole. The impregnated bag was supposed to cool the gases of explosion to a point where the firedamp would not be ignited

Ref: Daniel (1902), 150

Flameless Cartridge Cases (Ger & Amer). Compare with Combustible Cartridge Cases invented in Germany and mentioned in Vol **2**, p C78-R

Compositions of some combustible and semicombustible cases were described by Gen W.K. Gormley in Ordn **47**, 231–34(1962)

Some of compns developed at Picatinny Arsenal at the time of publication of Vol **2** of Encycl (1962) were classified

Flameless Gas-producing Charges. The devices, useful in coal mines or pressure actuated devices, contain NGu, GuN, Nitrodicyandiamidine, or Dicyandiamidine nitrate with 5–35% of the total charge a hypophosphite such as NaH_2PO_2, $Ca(H_2PO_2)$, or $NH_4H_2PO_2$. AN and alkali nitrates are added as needed. When fired with a gasless igniter or a cap, the compns are converted into permanent gases without flame at a temperature below that needed to ignite firedamp

Ref: A.T. Tyre, USP 2470082(1949) & CA **43**, 5190(1949)

Flameless Securites. These explosives were manufd at Denaby, England by Flameless Explosives Co, Ltd at the end of the 19th century. They were for use in gaseous mines and contained AN, DNB and AmmOx

Ref: Daniel (1902), p 711

Flamelessite. A mixt of nitrates with charcoal and other ingredients used at the end of the 19th century in England

Ref: Daniel (1902), 301

Flame or Light Accompanying Detonation. See under "Detonation (and Explosion) Luminosity (or Luminescence) Produced On" in Vol **4**, pp D425-L to D434-L

Flame, Miscellaneous Subjects are discussed in the following:

Refs: 1) H.G. Wolfhard & W.G. Parker, ProcPhysSoc(London) **62A**, 722–30(1949) & CA **44**, 1298(1950) (Examination of flames) 2) S.F. Boys & J. Corner, PrRoySoc **197A**, 90–106(1949) & CA **44**, 1784(1950)(Structure of the reaction zone in a flame) 3) J. Corner, PrRoySoc **198A**, 388–405(1949) & CA **44**, 2197(1950)(Effect of diffusion of the main reactants on flame speeds in gases) 4) T.H. Dimmock et al, US Dept Commerce, OfficeTechServ, **AD 262368**, 30 pp(1961) & CA **58**, 2925(1964)(The electrical props of ionized flames. Electrostatic and magnetohydrodynamic deflection) 5) R.J. Heinsohn & J.E. Lay, IAA Accession No **A65–13220**, 11 pp(1964)[From Intern Aerospace Abstr **5**(4), 542(1965)]& CA **62**, 12967(1965)(A flat flame under impressed electric and magnetic fields) 6) P. Laffitte et al, 10th SympCombstn **1964**(Pub 1965)(Engl) & CA **64**, 3275(1966)(Decompn flame of Hydrogen Azide) 7) H.M. Wight, NASA Accession No **N65–22812**, Rept No **U–2931**, 110 pp (1964) [From SciTech Aerospace Rept **3**(12), 2073(1965)]& CA **65**, 15142(1966)(Reflection and scattering of sound by flames) 8) J. Adler, CombstnFlame **9**(3), 273–79 (1965)(Engl) & CA **64**, 3275(1966)(Prediction of laminar flame speeds in stoichiometric mixtures with non-normal diffusion) 9) J. Debiesse et al, CR, Ser A, B **262B**(21), 1374–6(1966) & CA **65**, 8149(1966)(Analogies between the conductor props of a flame and those of a semiconductor) 10) A. Thomas & G.T. Williams, ProcRoySoc (London), Ser A **294**(1439), 449–66(1966) & CA **65**, 14457(1966)(Flame noise: sound emission from spark-ignited bubbles of combustible gas) 11) J.N. Bradley et al, CombstnFlame **10**(3), 259–66(1966) & CA **65**, 19920(1966)(Stabilized low temperature flames of acetaldehyde and propionaldehyde. A mass spectrometric study) 12) F.J. Weinberg, CombstnFlame **10**(3), 267–72 (1966) & CA **65**, 19920(1966)(Direct recording of flame ions on photographic emulsions)

Flame, Muzzle. See Muzzle Flash, under Flash Reducing Compounds

Flame Photography. See under "Flame Length and Duration Measurements"

Flame Photometry. An analytical method suitable for qual and quant detn of about 70 elements, flame photometry is based on the classical flame tests for the alkali and alkaline-earth metals (Na yellow, K purple, Ca brick-red, Sr carmine-red, etc). If a flame can be kept burning uniformly for an extended period of time and material fed into the flame at a const rate, the intensity of the spectral line or band will be a measure of the concn of the substance. The wave length of the emitted light will permit identification of the excited species

In addn to being the general term for the field as a whole, flame photometry refers also specifically to systems where the emitted light is separated by filters and the intensities are measured by a photo tube. Flame "spectrophotometry" uses a monochromator to resolve the light. Flame "spectrography" is emission spectrography using flame excitation and photographic recording
Refs: 1) F. Burriel-Marti & J. Ramirez-Muñoz, "Flame Photometry", Elsevier, NY (1957) 2) R. Hermann & C.T.J. Alkemade, "Chemical Analysis by Flame Photometry", 2nd Rev Edit, Trans by P.T. Gilbert, Jr, Interscience, NY (1963)

Flameproof (or Fireproof) Compounds are substances used to impregnate various flammable materials to make them fire-resistant or capable of burning without flame. Numerous compns are given in Refs 1 & 2. (See also Fire-Resistant Textiles and Fire-Retardant Paints)
Refs: 1) H. Bennet, "The Chemical Formulary", Vol **1–7**, Van Nostrand, NY (1933–37) 2) G. Hiscox & T.O'C. Sloane, "Fortunes in Formulas", Books Inc, NY (1947), 341–44

Flameproofing Substances. See Flameproof Compounds, Fire Resistant Textiles, and Fire Retardant Paints

Flameproofing of Textile Fabrics. See Fire Resistant Textiles

Flame Propagation and Velocity are described in the following:
Refs: 1) Ya.B. Zel'dovich, JPhysChem (USSR) **22**, 27–48 (1948) & CA **42**, 5229 (1948) (Theory of propagation of flame. Calcs rate of flame propagation for a specific relation between diffusion and heat conductance) 2) Ya.B. Zel'dovich, Natl-AdvisoryCommAeronaut, TechMemNo **1282**, 39 pp (1951) & CA **45**, 6844 (1951) (May be the same as Ref 1) 3) W. Jost, ZPhysikChem **196**, 298–304 (1950) & CA **45**, 8774 (1951) [The spreading of a flame from a spark is considered on the basis of the general theory of flame propagation. The results of tests are similar to those reported by B. Lewis & G. von Elbe in CA **42**, 762–63 (1950)] 4) A. Egerton & D. Sen, 4th-SympCombstn (1953), pp 321–28 (Flame Propagation: The Influence of Pressure on the Burning Velocities of Flat Flames) 5) R.A. Strechlow & J.G. Stuart, 4thSymp-Combstn (1953), pp 329–36 (An Improved Soap Bubble Method of Measuring Flame Velocities) 6) R. Sandri, CanJChem **34**, 313–23 (1956) & CA **50**, 12481 (1956) (Flame propagation in expl mixture of gases. I. general theory.) (Sets up and discusses the system of differential equations of flame propagation. Reduces the energy eq to the form of Abel's differential eq of the 2nd type and develops a simple numerical soln. Gives an approximation formula for the flame velocity which is useful in most practical cases of combustion) 7) V.K. Zotin & A.V. Talantov, IzvVyschikUchebnZavedenii, Aviats-Tekhn **9**(3), 98–103 (1966) (Russ) & CA **65**, 19919 (1966) (Flame propagation velocity in turbulent flow of homogeneous mixtures. Makes an analytical study of dependence of flame propagation velocity upon the pulsation velocity and upon the normal burning velocity. Obtains a relationship between turbulent flow propagation velocity and degree of preheating)

(See also "Épreuve de propagation dans une gouttière" in Vol 5 of Encycl, p E138-L and also some refs under "Flame Theories")

Flame Radiations are discussed in the following:

Refs: 1) Sh. Silverman, "The Determination of Flame Temperature by Infrared Radiation", 3rdSympCombstn (1949), 498–500 (8 refs) 2) A.G. Gaydon & H.G. Wolfhard, "Flames, Their Structure, Radiation and Temperature", Chapman & Hall, London (1953) and 2nd Edition of 1960 3) G. Monnot & M. Riviera, RevInstFrançPetroleAnnCombust-Liquides **19**(6), 803–37 (1964) & CA **61**, 14429 (1964) (Thermal radiation from liquid-fuel flames) 4) A.H. Lefebvre, Symp-(Int)Combstn, (Proc), 12th **14–20 Jul 1968**, 1247–53 (Pub 1969) (Radiation from flames in gas turbines and rocket engines, a review with 25 refs) & CA **75**, 38323 (1971) 5) V. Pikashov et al, Inzh-FizZh **20**(2), 352–3 (1971) & CA **75**, 40700 (1971) (Determination of the temperature and radiation characteristics of a flame by changing beam length)

Flame Reactions and Detonations (Flammenreaktionen und Detonationen, in Ger). Title of the Symposium (Diskussionstagung) held under the auspices of the Deutsche Bunsen-Gesellschaft für physikalische Chemie at Troisdorf, Germany from 18 to 20 October, 1956

The report (Bericht) of the symposium is published in the Zeitschrift für Elektrochemie, Berichte der Bunsengesellschaft für physikalische Chemie, vol 61, No 5, 1957, Verlag Chemie-GMBH-Weinheim/Bergstr, pages 559–692

The report includes the following papers:

1) W. Jost, Flammenreaktionen und Detonationen (Flame reactions and detonations), Introduction, pp 559–62
2) C. Franze & H.Gg. Wagner, Theorie der Flammenausbreitung (Theory of flame propagation), p 562–4
3) J.H. Burgoyne & F.J. Weinberg, Studies of the Mechanism of Flame Propagation in Premixed Gases, pp 565–69
4) J.E. Dove et al, Flame Propagation in Carbon Monoxide–Oxygen Mixtures, pp 570–74
5) B. Lewis & G. von Elbe, Fundamental Principles of Flammability and Ignition, pp 574–78
6) A. van Tiggelen et al, Eine Beziehung für die Flammengeschwindigkeiten verschiedener Brennstoffgemische (A correlation for velocities of flame propagation of various combustible mixtures), pp 579–83
7) Sir A.C. Egerton & K.K. Roy, The Oxidation of Weak Methane Mixtures at High Temperature, pp 584–85
8) N. Manson, La Théorie Hydrodynamique et le Diamètre Limite de Propagation des Ondes Explosives (Hydrodynamic theory and the maximum limiting diameter of propagation of explosion waves), pp 586–92
9) F.C. Harschbarger, Shock Tube Technique, pp 592–600
10) R.F. Simmons & H. Wolfhard, The Light Emission of Halogen Flames, pp 601–09
11) H. Behrens & F. Rössler, Temperaturmessungen an Russ-strahlen in Flammen (Temperature measurements on jets of carbon black in flames), pp 610–13
12) A. Berthmann, Untersuchungen über den Detonationsvorgang gewerblicher Sprengstoffe hinsichtlich ihrer Zusammensetzung, Prüfung und ihrer Wirkung in der Praxis (Investigation of the detonation process of industrial expls with regard to their compn, testing and their effect in practice), pp 614–28
13) R. Schall, Methoden und Ergebnisse der Detonationsdruckbestimmung bei festen Sprengstoffen (Methods and results of the determination of blast pressure of solid explosives), pp 629–35
14) H. Ahrens & E. Eitz, Röntgenblitzaufnahmen zur Untersuchung der Detonationsübertragung bei Wettersprengstoffen (X-ray flash photographs for the investigation of the transmission of detonation of safety mining explosives), pp 635–42
15) J.F. Roth, Versuche mit einem Stosspendel zur Aufklärung des Detonationsablaufes bei Wettersprengstoffen (Investigations with an impact pendulum for

clarifying the detonation path in safety mining explosives), pp 643–51
16) H. Käufer, Untersuchung der Flammenentwicklung bei Sprengstoffdetonationen und der dadurch hervorgerufenen Zündungen eines Methan-Luft-Gemisches bei verschiedenen Schussanordnungen mittels Aufnahmen auf rotierendem Film (Investigation by means of a rotating drum camera of flames evolving from detonations of explosives at various shooting arrangements). These flames cause the ignition of a methane-air mixture), pp 651–62
17) H. Freiwald & H. Ude, Untersuchungen an kugelförmigen Detonationswellen in Gasgemischen (Investigations of spherical detonation waves in gas mixtures), pp 663–72
18) H. Selle, Deflagrations-und Detonationserscheinungen beim Zerfall von Methylnitrit (Deflagration and detonation phenomena during the decomposition of methyl nitrite), pp 672–78
19) Th. Just & H.Gg. Wagner, Gleichgewichtseinstellung in Gasdetonationen (The reaching of equilibrium in the detonation of gases), pp 678–85
20) K. Fischer, Ablauf von Kohlenstaubexplosionen (The process of coal dust explosions), pp 685–92
(See also Vol **4**, p D348-L, Detonation, Flame Reactions and)

Flame Spectra of Explosives are discussed in the following:
Refs: 1) H. Behrens & F. Rössler, Naturwissenschaften **36**, 218(1949) & CA **44**, 3798(1950)(Flame spectra with nitrogen oxide as combustion partner) 2) H. Behrens, ZElektrochem **54**, 535–38(1950) & CA **45**, 5020(1951)(Spectra of nitrous oxide flames and reaction kinetics) 3) H. Behrens & F. Rössler, ZNaturforsch **6a**, 154–60(1951) & CA **45**, 8361(1951)(Flame spectra of systems with NO_2–bound oxygen) 4) P. Ausloos & A. van Tiggelen, "Quantitative Spectrographic Investigation of Flames", 4thSympCombstn(1953), pp 252–59(10 refs) 5) H.A. Bent & B. Crawford, JPhysChem **63**, 941(1959)(Infrared studies of propellant flames) 6) H.W. Thompson, Editor, Advances in Spectroscopy **2**, 23–56 (by A.G. Gaydon), Interscience, NY(1961) 7) H. Selzer, RaketentechRaumfahrtforsch **7**(2), 41–6(1963) & CA **61**, 10523(1964) (Combustion mechanism of composite propellants) 8) D. Naudeix, RevGenTherm **10**, 115–16, 643–60(1971) & CA **75**, 145833 (1971)(Emission spectra of gas flames) [See also under Detonation (and Explosion), Luminosity (Luminescence) Produced On" in Vol **4** of Encycl, pp D425-L to D434-L]

Flame Temperature Determinations are described in the following:
Refs: 1) Sh. Silverman, "The Determination of Flame Temperatures by Infrared Radiation" 3rdSympCombstn(1949), 498–500(8 refs) 2) J.A. Curcio et al, JOptSocAmer **41**, 173–79(1951)(A method for the determination of flame temperature from emission in the ultraviolet OH band) 3) A.G. Gaydon & H.G. Wolfhard, "Flames, Their Structure, Radiation and Temperature", Chapman & Hall, London(1953) and 2nd Edition(1960) 4) A.S. Leah & N. Carpenter, "The Estimation of Atomic Oxygen in Open Flames and the Measurement of Temperature", 4thSympCombstn(1953), pp 274–85(27 refs) 5) M. Gilbert & J.H. Lobdell, "Resistance Thermometer Measurements in a Low Pressure Flame", 4thSympCombstn(1953), pp 285–94(18 refs) 6) A.M. Ball, "Solid Propellants", Part **1**, **ORDP 20–175**(1961), pp 6–7 & **AMCP 706–175**(1964), pp 6–7

Flame Temperature of Priming Mixture. The flame temperature of a priming mixture can be calculated, by assuming reactions at room temperature and the resulting products then being heated to the adiabatic flame temperature, by the expression:

$$T_{flame} = T_{initial} + \frac{Qv - \Sigma Lp}{\Sigma Cp}$$

where: Qv = heat of reaction at constant volume
ΣLp = sum of the latent heats of fusion of the products
ΣCp = heat capacities of the products

For purposes of this calculation, latent heats at constant volume and at constant pressure are assumed equal, heat capacities at constant pressure and at constant volume are assumed equal for solids and liquids
[See also "Calculation of Temperature of Detonation (and Explosion)" and "Experimental Determination of Temperature of Detonation (and Explosion)", under "Detonation (and Explosion) Temperature Developed On" in Vol 4 of Encycl, pp D589-L to D601-R]

Flame Test for Detonators. This test, designed by Dr Grave, consists of photographing the flash produced by a detonator when exploded. The test is practically valueless because the flame is of nearly the same intensity for different detonators, varying only in size with increase or decrease of the detonator charge

Flame Tests. See "Flame, Length and Duration of", "Flame Photometry", "Flame Propagation and Velocity" and "Flame Temperature Measurements", in this Vol

Also in Vol 1: "Flame Test", p XII; "Index of Inflammability Test", p XVII and "Sen sitivity to Flame, Heat, Sparks, Electrostatic Discharges, etc", pp XII & XXIII

French Official Tests 5 & 6 are described in Vol 5 under "ESSAIS d'EXPLOSIFS", p E138. They are: a) "Combustion en gouttière de 20mm" (Combustion in a 20mm Trough), also known as "Aptitude à l'inflammation" (Capability to Inflame) and b) "Combustion en tas conique" (Combustion in a Conical Pile), also known as "Épreuve de sensibilité à l'inflammation" (Test for Sensitivity to Inflammation)

Flame Theories. The following are some refs on this subject:
Refs: 1) Ya.B. Zel'dovich & D.A. Frank-Kamenetskii, ActaPhysicochim (Russia) **9**, 341–50 (1938) (in Engl) & CA **33**, 7570 (1939) (A theory of thermal propagation of flames) 2) B. Lewis & G. von Elbe, JChemPhys **15**, 803–08 (1947) & CA **42**, 762–63 (1948) (Theory of the propagation of flame from an instantaneous point source of ignition) 3) Ya.B. Zel'dovich, ZhFizKhim **22**, 27–48 (1948) & CA **42**, 5229 (1948) (Theory of propagation of flame. States conditions in an expl chem reaction necesssry for propagation of the flame at a const rate. Calcs this rate for a definite relationship between diffusion and heat conductance. Evaluates the effect of chain reactions on the propagation of the flame) 4) B. Karlovitz, JChemPhys **19**, 541–46 (1951) & CA **45**, 9341 (1951) (Theory of turbulent flames) 5) G. Klein, Phil-TransRoySocLondon **249**, 389–415 (1957) & CA **51**, 7114 (1957) (Flame theory) 6) D.B. Spaulding, 6thSympCombstn, Yale Univ **1956**, 12–20, 35 refs (Publ 1957) (Ends and means in flame theory, a review) 7) B.F. Gray, TransFaradaySoc **65** (6), 1603–13 (1969) & CA **71**, 14732 (1969) (Unified theory of explns, cool flames, and two-stage ignitions) 8) C-H. Yang & B.F. Gray, TransFaradaySoc **65** (6), 1614–22 (1969) (Unified theory of explns, cool flames, and two-stage ignitions)
(See also "Combustion Theories as Applied to Solid Propellants" in Vol 3 of Encycl, pp C430-L to C433-L)

FLAME THROWERS (Flame Projectors) are devices for throwing a spray of combustible liquids, gels or gas a considerable distance, with the primary object of burning enemy personnel and sometimes of igniting easily inflammable material of the enemy

The origin of gas flame throwers may be traced to the 5th century BC (See under Incendiary Agents), but the liquid flame throwers were invented in about the 7th century for use in conjunction with "Greek Fire". With the invention of gunpowder and guns, flame throwers were forgotten until the Japanese reintroduced them during the Russo-Japanese War of 1904–1905. The Germans worked on their perfection and by 1912 had developed a model which, adopted as standard equipment for the German Army, was used during WWI and served as a prototype for other German, as well as British, French, Italian,

and later, American models of WWI (Refs 1 & 6)

The principle of all flame throwers is practically the same: a flammable liquid or gel contained in a strong metal reservoir is put under heavy pressure by a compressed gas (such as air, CO_2, N_2 or H_2) called propellant, contained in a steel cylinder connected to the reservoir thru a reducing valve, designed to maintain a given pressure in the reservoir. The liquid is forced out of the reservoir thru a pipe fitted with a valve and terminating with a nozzle, of suitable diameter, which directs the stream in the direction desired. The liquid is ignited either at the end of the nozzle by means of a special igniter or, where it is projected onto the earth, by means of incendiary grenades which are thrown into the area reached by the stream

The ignition of projected liquids is effected either by a hydrogen pilot lamp or by a cartridge of a slow-burning, combustible-oxidizing agent mixture attached to the nozzle and ignited by an electric detonator or by friction (Ref 1)

During WWI two kinds of flame projectors were used:

1) *Stationary type* – large devices, holding up to about 100 gallons of liquid and used for trench defenses

2) *Portable type* – small devices holding 2–5 gals of liquid, which could be carried on the backs of the operators by means of suitable straps and pads. They threw liquid from 60 to 125 ft and could be emptied of liquid by continuous discharge in less than 20 seconds

According to Ray (Ref 1), the following projectors were used during WWI:

1) *Stationary type:*

a) German *Flammenwerfer* contained 45 gals of liquid, which was propelled by compressed nitrogen

b) French L-1- held 33 gals of liquid, which could be projected by means of compressed air up to 180 feet; the duration of projection was 15 seconds

c) *British Stationary Projector* held 80 gals of liquid, which was projected 300 ft by means of compressed "deoxygenated air"

d) USA. No stationary machine was developed by the US Armed Forces because the usefulness of such machines was questioned

2) The following *Portable Type Flame Projectors* were developed during WWI:

Table F5

Designation	Wt Charged, lbs	Capacity gals	Propellant	Working Pressure psi	Nozzle Diam inch	Range of Projection ft
German "Wex"	66	2.5	Nitrogen	288	Inter-changeable	80–100
French P–3	53	3.0	Air	170	0.24	98
Italian DLF	40	1.5	Air	–	–	83
British, Lawrence	65–70	3.0–3.5	CO_2	240	0.36–0.48	125
American, Boyd No 3	70	4.75	H_2	240	0.31	115

For composition of liquids used in these and other projectors, see further under "Flame Throwers-Liquids"

Mechanized or Tank-type Flame Throwers. During WWII a 3rd type of flame thrower was developed, the so-called *"tank-mounted"* or *"mechanized"* flame thrower, which at first was intermediate in size between the stationary and portable types but later surpassed the WWI stationary types in size. Although tank-mounted throwers were effective at targets over 200 ft distance, the best results were obtained when operated at extremely close range. The tank, being protected by armor, could approach an enemy's fortification and fill it, thru an embrasure, with flame and smoke, blinding, burning and shocking enemy personnel

The following may be given as examples of tank-type flame throwers:
a) *British Mechanized Thrower* carried by the 41-ton Churchill "Crocodile" tank, threw a geyser of fire over 450 feet and could be reloaded during action because the tank towed its own fuel in an armored trailer
b) *American Mechanized Thrower*, Model E-9 had a range up to 600 ft and a nozzle diameter of about ¾ inch. The fuel was 10% *Napalm* and was carried in a trailer tank (800–1200 gals capacity) attached by a joint to the M-5A1 tank, in which a gun was mounted. The fuel was ejected by means of compressors at a pressure of 500 psi
Portable Flame Throwers of WWII. There was no progress in the construction of flame throwers between the two wars, consequently, when WWII started, the only available models were those used in WWI. In 1941 work was started in the USA and by 1942 several models of portable throwers were constructed; all of them using one of the newly developed thickened fuels, such as "Gelgas" of the Standard Oil Co (Esso) or "Napalm", developed at Harvard University. Flame throwers using "Gelgas" were sometimes called "Essothrowers". The principal ones developed by the US Army were models M-1A1 and M-2, both using Napalm thickened fuel (4.2p of Napalm in 95.8p of ordinary gasoline). In the earlier models ignition of fuel (as it emerged from the ignition head) was done by means of electric sparks, but as this method was reported to be unreliable when used in tropical countries, a new ignition system was developed in which ignition of the fuel was accomplished by flame producing pyrotechnic cartridges

The later US portable unit carried and fired by one man is described by Fisher (Ref 6, p 51). It weighs 70 lbs and consists of two interconnected fuel tanks (2 gals capacity each) with a pressure tank (containing either nitrogen or air) placed between them. The pressure of the gas in the middle tank is such that it can eject the fuel with a pressure of 2000 psi. The so-called "gun system", which resembles a Browning pistol in appearance, consists of a trigger, an ignition head, and a hose to convey fuel from the tank to the gun assembly. Before reaching the gun, the fuel must pass thru a valve, which has to be opened by pressure on the "valve lever". Pressure on the gun trigger activates the ignition device described above. The contents of the tank last only 10 seconds, if ejected in one application. Much better results are obtained by firing the thrower in a series of bursts of 2–3 seconds each. The effective projectory range is up to 125 ft. After firing, the tanks can be refilled for further use

Towards the end of WWII, in 1945, some very light portable throwers were developed, which were *"one-shot"* types intended to be discarded after use (Ref 6)
Refs: See below under Flame Throwers–Liquids and Gels

Flame Throwers–Liquids and Gels. One of the original liquids (or semi-liquid, semi-solid) for flame throwers was the so-called "Greek Fire" invented in the 7th century

The liquids used during WWI and WWII were combinations of heavy and light distillates of petroleum, coal tar or wood tar. The presence of small amounts of light distillates is necessary in order to secure easier ignition, while the presence of heavy distillates is necessary because heavy liquids can be thrown farther and are longer-burning

The British, French & Italians during WWII used mixtures of heavy and light petroleum distillates with a density of about 0.86 at 15°. The Germans used various mixtures of petroleum distillates, coal tar fractions, and sometimes such liquids as methanol, acetone or even ether. The density of such mixtures was usually about 0.96 at 15°. One of the first American mixtures contained 70% water-gas tar (flash point 122° and d 1.044) and 30% "benzene heads" (fl p 26° and d 0.756). The resulting mixture had d 1.02. It gave a good trajectory, fierce flame and good throw. Crude benzene was later substituted for the "benzene heads" as being more readily available. In cold weather the amount of benzene could be increased to 40%

Because flammable liquid materials are not convenient to handle, attempts were made to convert them into a solid state. During WWI solid oils had already been prepd by treating the petroleum distillates with sodium stearate or other materials, but they were used only in some incendiary bombs, shells, Liven's drums and trench stoves, and not in flame throwers

At the beginning of WWII, materials used in flame throwers were liquids, which were no better than those used during WWI

In 1941, exploratory work on incendiary materials was begun under L.F. Fieser at Harvard University (Ref 7). The first material developed was gasoline (80-octane motor-vehicle type), thickened (jellied) by addition of natural rubber. This was a satisfactory product but, due to the shortage of natural rubber, it could not be produced on a large scale. Therefore, a new thickener was developed which consisted of crude aluminum naphthanate modified by addition of Al soaps of cocoanut oil acids. This thickened gasoline was not very successful at first (See further). Standard Oil Co (Esso) meanwhile developed a very satisfactory thickener, called **gelgas** or **jellied gasoline** based on "isobutyl methacrylate polymer" which was adopted by the US Armed Forces as fuel for special flame throwers, called **Essothrowers.** However, the work on naphthenates was continued and success was achieved when purified aluminum naphthenate (by extraction with alcohol or acetone) replaced the impure material. By combining the aluminum salts of naphthenic, oleic and cocoanut acids, a product called **Napalm** was obtained. This was produced by aqueous coprecipitation with $Al_2(SO_4)_3$ solution of the mixed sodium soaps of naphthenic, oleic and cocoanut oil acids in the ratio 1:1:2. The resulting precipitate was filtered, washed with water and dried. It was a granular,non-agglomerating powder which was reasonably resistant to oxidation. This powder could be easily dissolved, by stirring at room temperature, in gasoline or other petroleum hydrocarbons and used, not only in flame throwers, but also in incendiary bombs of different sizes

For portable American throwers, such as M-1A1 or M-2, 4.2g of Napalm were dissolved in 95.8g of ordinary gasoline at a temp above 15.5°(60°F). For "mechanized" (tank) flame throwers, as for instance Model E9, a 10% solution of Napalm was used

It should be noted that Napalm gels were better suited for flame throwers than any of the existing thickened fuels. Gels based on the isobutylmethacrylate polymer, while very suitable for loading incendiary bombs, when used in flame throwers had a tendency not to issue from the nozzle in a continuous (uninterrupted) stream, as the Napalm gels do, but to pull apart into separate small chunks. This offered so great a surface that drag, owing to air resistance, reduced the range. It can be said that "Esso" gels lack "stringiness" or are "short"

Another advantage of Napalm gels lies in the fact that they are anomalous (pseudoplastic) in their flow characteristics. This means that these gels do not obey the "Newtonian" laws of flow for ordinary liquids, such as lubricating oils. When these (Newtonian) liquids are caused to flow thru a narrow tube or orifice, under pressure, the flow is directly proportional to the pressure as long as the turbulent flow range is not reached, meaning that if the pressure is increased twice, the flow is also doubled. This is not true for Napalm gels, in which the rate of flow increases much faster than the increase in pressure. For example, with a 6% Napalm gel, an increase of pressure equal to 25% increased the rate of flow thru a 1½ inch pipe nearly a thousand-fold (Ref 7)

During WWII, the British used a thickened fuel called *Fras* which was prepared by digestion of aluminum stearate with gasoline at 50–55°(120–130°F), but this material was not as good as Napalm and more difficult to prepare (Ref 7)

It should be noted that the use of thickened fuels not only solved the problem of easier handling, but it also increased the throwing range as well and produced a more regular trajectory. In addition, due to the fact that the gel easily adheres to the objects it hits (or even envelopes them) and does not splash as liquids do, a hotter and more concentrated flame is produced

By using these fuels, it was possible to fire "around a corner", meaning that the stream of jelly can be directed on the fortification from the flank out of range of enemy guns. After striking the sides of the gun port, the jelly spatters inside the fortification, burning the gun crew and suffocating them by using up the oxygen present

For filling incendiary bombs, a thicker solution was used than for flame throwers. For instance, for M-69, small incendiary bomb, the following composition was used: Napalm thickener 9 to 12, gasoline 91 to 88. *Pyrogel* or *"Goop":* Sticky and doughy gray mass, also called "synthetic lava" was prepd by mixing jellied gasoline with "crude magnesium" powder, liquid asphalt & petroleum oils. The crude magnesium was obtained as a residue from production of high grade magnesium by the Hansgirg process. Pyrogel was used in the so-called M-76 "Goop" bomb, also called "Block burner", described under Incendiary Warfare. This mixture combined the properties of jellied gasoline and magnesium as follows: a) it spouts flame like gasoline; b) emits very intense radiant heat of a burning metal; c) burns downward as well as upward and d) continues to burn and flare up for a considerable period of time; e) sticks to objects and penetrates into hard-to-reach corners of structures

Refs on Flame Throwers: 1) A.B. Ray, IEC **13**, pp 720–22(1921) 2) A. Prentiss, "Chemicals in War", McGraw-Hill, NY(1937), p 260 3) C. Wachtell, "Chemical Warfare", ChemCatCo, Brooklyn(1941), p 265 4) R. Grant, "Sixty-yard Flash", Popular Mechanics, Nov(1944), pp 82–86 & 150–2 5) V. Torrey, "New Incendiary Bombs Packed with Gel-Gas", Popular Science, May(1945), pp 100–109 5a) L.F. Fieser et al, "Napalm", IEC **38**, 768–73(1946) 6) G.B. Fisher, "Incendiary Warfare", McGraw-Hill, NY(1946), pp 35, 50–53 & 103–105 7) W.A. Noyes, Jr, Editor, Chemistry(Science in WWII), Little, Brown & Co, Boston(1948), pp 388, 389 & 410–22 8) J.E. Burchard, "Rockets, Guns and Targets", Little, Brown & Co, Boston(1948), p 74 9) War Department Technical Manuals on Flamethrowers (unclassified): a) TM 3–375. Portable Flame Throwers M1 and M1A1(1943); b) TM 3–378. Manifold, Portable Flame Thrower, E4(1945); c) TM 3–362. Flame Thrower, Mechanized, M3-4-3(Installed in Medium Tanks M4A1 & M4A3(1945); d) TM 3–363. Flame Thrower, Mechanized, E5R2-M3(1945); e) TM 3–376A. Portable Flame Thrower, M2-2(1944) 10) K.J. Mysels, IEC **41**, 1435–38(1949)(Preparation and props of Napalm) 11) Kirk & Othmer **6**(1951), 578–81; 2nd Editn **4**(1964), pp 889–94, by J.N. Bruce (under Chemical Warfare

Additional Reference on Flame Throwers and Projectors: A) E. Neumann, "Zur Entwicklungsgeschichte des Flammenwerfers", SS **14**, 105–06 & 138–43(1919) B) A. Hausenstein, SS **34**, 10–14, 44–7, 73–7, 105–09 (1939) & CA **33**, 5661(1939)(Historical discussion on the use of fire in warfare) C) H.C. Hottel, OSRD **615**(PBL **23769**(1942) (Studies on fuel projection from nozzles) D) H.C. Hottel & G.H. Garaway, OSRD **637** (PBL **32717**)(1942)(Joint report on status of NDRC projects on flamethrowers) E) Standard Oil Development Co, OSRD **983** (PBL **4188**)(1942)(Development of Flamethrowers) F) E.C. Kirkpatrick, OSRD **3507** (PBA **4627**)(1944)(Chemical ignition of flame throwers) G) Standard Oil Development Co, OSRD **3574**(PBL **4187**)(1944) (Development of Portable Flamethrower E2) H) M. Morgan, OSRD **1346**(PBL **5968**)(1945) (Development of Morgan-type Flametrrower for M4A1 Tank) I) A. Byfield & others, OSRD **6190**(PBL **4190**)(1945)(Flamethrowers, incendiaries and their evaluation) J) E.E. Bauer & E.K. Carver, OSRD **6236** (PBL **28628**)(1945) K) Standard Oil Development Co, OSRD **6376**(PBL **22224**)(1945) (Development of flamethrowers, service units and thickened fuels) L) C.M. Cawley et al, JInstPetroleum **33**, 721–55(1947) & CA **42**, 3572(1948)(Flamethrower fuels contg Al stearate gels) M) Anon, "M2A1 Portable Flamethrower", **TM 3–376**(1952) N) Anon, "Flamethrowers and Fire Bomb Fuels", **TM 3–366**(1958)

Flamethrower's Mixtures–Gel Type. The following Table F6, reproduced from Ref, gives compns of mixts developed in the USA between 1941–1945, incl

Ref: A.E. Gaul & L. Finkelstein, "History of R & D of the CWS in WWII", 1 July 1941 to 31 December 1945, Vol 18, Part IV (1949), p 662

Table F6

Composition of Flamethrower Mixtures – Gel Type

	IM–1	Napalm	PT–1	PT–2	N–1	N–2	N–3	N–4	N–5	N–6	N–7	N–8	N–9
Stearic acid	3.0	–	–	1.0	–	–	–	–	–	–	–	–	–
Isobutyl methacrylate polymer AE	5.0	–	–	–	–	–	–	–	–	–	–	–	–
Calcium oxide	2.0	–	–	–	–	–	–	–	–	–	–	–	–
Gasoline	88.75	86.5	–	19.25	–	–	–	–	–	–	28.5	–	–
Water	1.25	–	–	–	–	–	–	–	–	–	–	–	–
Napalm polymer	–	13.5	–	–	–	–	–	–	–	–	–	–	–
60/40 Permanent goop	–	–	49.00	45.00	–	–	–	–	–	–	–	–	–
Magnesium, "S" chips	–	–	10.00	10.00	20.0	20.0	20.0	18.0	5.0	30.0	19.0	25.0	20.0
Sodium nitrate (coarse)	–	–	4.75	4.75	–	–	–	–	–	–	–	–	–
AE polymer	–	–	3.00	–	–	–	–	–	–	–	–	–	–
B SE/MNE 50/50	–	–	3.00	–	–	–	–	–	–	–	–	–	–
15% GR-S in SR gasoline	–	–	–	20.00	–	–	–	–	–	–	–	–	–
S_2Cl_2 in 10% solution in WW kerosene	–	–	–	–	–	–	–	–	–	0.2	–	–	–
S_2Cl_2 in SR gasoline	–	–	–	0.25	0.25	0.25	0.25	0.25	0.25	–	2.0	1.0	1.0
24% GR-S in SR gasoline	–	–	–	–	60.0	52.0	50.0	50.0	50.0	52.0	6.0	49.0	59.0
Barium nitrate	–	–	–	–	8.0	12.0	15.0	15.0	20.0	18.0	–	–	12.0
Magnesium (fine, B grade)	–	–	–	–	–	16.0	15.0	12.0	20.0	–	9.5	6.0	8.0
Sulfur	–	–	–	–	–	–	–	5.0	5.0	–	5.0	4.0	–
SAE No 10	–	–	–	–	–	–	–	–	–	–	6.0	2.0	–
Ammonium perchlorate	–	–	–	–	–	–	–	–	–	–	24.00	13.0	–

Flame Thrower–M132A (Amer). A self-propelled device (designed and manufd at Edgewood Arsenal, Md) which is capable of throwing a steady stream of flame more than 150 yards. Operated by a two-man crew, the thrower can fire continuously for 30 seconds or may be fired in bursts
Ref: Anon, Ordnance, July-August 1968, p 30-L

Flamethrower, Portable for Lighting Fuses. See Vol 4 of Encycl, p D762-L

Flaming Bayonet, invented during WWI, was a flame throwing type of attachment for the muzzle of a military rifle. It was for use in bayonet fighting and consisted of either a small (less than 1 lb) charge of flammable liquid or of a cartridge contg a flash compn. When the bayonet hit a target, a spurt of flame 10–30 ft in length was produced. The cartridge type was considered superior to the liq type
Ref: Anon, "History of Trench Warfare Materiel", Army Ordnance Handbook No 154, GovtPrintgOff, Wash, DC (1920), pp 217–18

Flaming Thermit. An incendiary mixture developed by the British during WWI and used in their small unit (cluster) bombs. It was a mixture of commercial thermit and barium nitrate

Flammability. Its definition and brief description are given in Vol 5, pp D211-R to D213-R under "Detonability and Flammability of Explosives, Propellants and Pyrotechnic Compositions"

Flammability of Black Powder Igniter Bag by firing in a manometric bomb is described by A. Douillet & P. Miaud in MP 36, 277–84 (1954) & CA 50, 3764 (1956). The same technique was used as described by the same investigators in MP 34, 221–36 (1952) & CA 48, 4838 (1954)
(See also under Flammability of Propellants)

Flammability Characteristics of Combustible Gases and Vapors discussed by M.G. Zabetakis in USBurMinesBull 627 (1965), listed in Vol 5 of Encycl, p D348-L, bottom

Flammability and Explosibility of Dusts. Various tests are described under "Burning (Combustion) and Deflagration of Gases, Vapors and Dusts" in Vol 4 of Encycl, pp D154-L to D163-L. Also "Dust Explosibility" and "Dust Explosions" in Vol 5, pp D1578-L to D1579-R

Flammability and Explosibility of Gases and Vapors. See above item

Flammability of Explosives, Propellants and Pyrotechnic Compositions. See under Detonability and Flammability of Explosives, Propellants and Pyrotechnic Compositions in Vol 4 of Encycl, pp D211-R to D213-R

Flammability and Ignition is discussed in the book of S.S. Penner & B.P. Mullins, "Explosions, Detonations, Flammability and Ignition", Pergamon Press, NY (1959), listed in Vol 5 of Encycl, p D348-R, top

Flammability Index. See "Index of Flammability" in Vol 1 of Encycl, p XVII and in AMCP 706–177 (1967), p 3

Flammability Limits of Methanol-Air Mixtures: The Effect of Water Vapor On, is discussed by K. Dvořák & A. Reiser in ChemListy 49, 467–72 (1955) & CA 49, 9927 (1955) (A static app suitable for liquid fuels is described. Results for water-methane-air mixts are given. A simple correlation method on the basis of heat balances is attempted)

Flammability of Nitrocellulose. Highly dangerous in the dry state when exposed to heat, flame or powerful oxidizers. When wet with 35% denatured alc, it is about as hazardous

as ethyl alcohol or gasoline. Dry NC burns rapidly with intense heat and ignites very easily
Ref: Sax, 3rd edit(1968), p 965-R

Flammability of Plastics was discussed by W.J. Sauber & G.A. Patton in SPE(Society of Plastics Engineers) TechPapers **5**, No 63 (1959) & CA **53**, 10827(1959)(Critical view of methods for measuring flammability of plastics)

Flammability of Powders. A German apparatus for measuring the "Entzündungsfähigkeit des Pulvers" was described by N.L. Hansen in SS **2**, 165–67(1913)

Flammability of Propellant. The flammability of French propellants "poudre B" and "poudre Néo" were detd in high pressure bombs, by measuring the time from firing to rise of pressure due to ignition by Black Powder
Refs: 1) A. Douillet & P. Miaud, MP **34**, 221–36(1952) & CA **48**, 4838(1954) 2) A. Douillet & P. Miaud, MP **36**, 277–84(1954) & CA **50**, 3764(1956)

Flammability Ranges and Spontaneous Ignition of Some Fuels in Air were detd by J.H. Burgoine & R.F. Neale in Fuel **32**, 5–27(1953) & CA **47**, 2577–79(1953)

Flammability, Surface, of Materials: Measuring It by Using a Radiant Energy Source was described by A.F. Robertson et al in ASTM **1956**, Preprint 87, 17pp & CA **50**, 13443(1956)

Flammability Test for Thin Flexible Materials is described giving details of construction of apparatus in the British Standards Institute, Brit Standard **476**(1955), Pt 2, 11pp & CA **50**, 7462(1956)

Flammable Inhibitors Can Prevent Explosions in Chemical Plants. In a system where a combustible and oxygen (from air or added pure) are to be reacted and the operating conditions require an expl mixt and temps near the ignition temp, the possibility of expln can be eliminated by adding a diluent to the system. Diluting with combustible or oxygen may upset the stoichiometry of the system, and use of a sufficient quantity of inert gas may make the process uneconomical. A relatively small amount of a hydrocarbon (CH_4,etc) has been found to be as effective as a much larger amount of an inert gas in diluting the mixture. It is essential to stay below the lower expl limits of the hydrocarbon and oxygen, obviously
Ref: E. Jones, ChemEngr **59**, 185–8(1952)

Flammable Mass. A mass useful for making sparklers, etc is made by adding to a mixt of Al powder, KNO_3 and(or) $KClO_3$ and rice starch or dry powd CaCO . Perfume or incense may be added to the mass which burns down with fogging and formation of snow-like flakes
Ref: N. Wimmer, AustrianP 178599(1954) & CA **48**, 8544(1954)

Flammable Materials, Storage and Handling. Since it is not economically feasible to store each item of inventory under ideally safe conditions, a compromise must be made between perfect safety and industrial economy. Standard works such as those listed in the Refs should be consulted for general guidance and for information on specific points
Refs: 1) Factory Mutual System, "Handbook of Industrial Loss Prevention", 2ndEd, McGraw-Hill, NY(1967)(Consult the book's index under storage; there is no chapter devoted to storage as such) 2) Sax (1968), pp 208–26 discusses storage and handling in general, and pp 365–1251 is an alphabetical list of specific substances
3) National Fire Codes. An annual set of 10 vols published by National Fire Protection Association (NFPA): Vol **1**, "Flammable Liquids"; Vol **2**, "Dusts"; and Vol **3**, "Combustible Solids, Dusts, and Explosives" are most pertinent to this topic

Flammbombe. A German incendiary bomb of WWII contg an oil mixture and a HE bursting charge. The following types are described in Ref 1 and listed in Ref 2:

a) Flam C 250A (B or C) – contd 50kg of oil incendiary mixt and TNT bursting charge

b) Flam KC250 – same filling as above

c) Flam C500 – contd the incendiary oil consisting of 70% petroleum with 30% TNT dissolved and TNT bursting charge

Refs: 1) Anon, **TM 9–1985–2**(1953), pp 52–4 2) Fedoroff et al, PATR **2510**(1958), p Ger 49-L

Flammenauslöschenzusatz. Ger for "Flame Extinguishing Addition", described here under "Flash Reducing Compounds"

Flammivores. A series of Belgian permissible expls manufd from 1887 on by Société Anonyme des Poudres et Dynamites d'Arendonck. Several formulations are given in the Table F7

Refs: 1) Daniel (1902), p 301 2) Marshall **1** (1917), p 396 3) Naoúm, NG(1928), p 439 4) Pepin-Lehalleur(1935), p 419 5) Clift & Fedoroff **2**(1943), p F1 6) Anon, Explosifs (Belg), No **4**(1958), p 66

(See also Vol **2** of Encycl, p B29-R)

Flammocite. An explosive which was not allowed as a permissible in France but was used in Belgium under the name of *Flammivore 3-bis:* NG 6, AN 44, $AmmSO_4$ 5, Sn 14, NaCl 16, TNT 10, cellulose 5

Ref: E. Audubert, Chim & Ind (Paris) **10**, 929 (1923) & CA **18**, 471 (1924)

Flanschgeschoss (Ger) (Flange Projectile), also called "Squeeze-bore", or "Littlejohn" was a subcaliber projectile provided with a a flange and three hollow studs as shown on Figure and described in the TM 9–1985–3, p 360

It was fired from a cylindrical rifled barrel to which a smooth-bored, tapered muzzle extention was attached

The principal advantage of the "flange" projectile in comparison to the other subcaliber projectiles was that it had no parts to be discarded, because the hollow stud and the flange were easily depressed when the projectile passed from the rifled section of the gun to the smaller caliber smooth bore extension

(Compare with Arrowhead Projectile, Arrow or Needle Projectile, Disintegrating Band Projectile, Röchling Projectile, Sabot Projectile and Tapered Bore Projectile)

Ref: PATR **2510**(1958), p Ger 49

Table F7

(Flammivores)

	Ref 1	Ref 2	Ref 3		Flammivore			
			No 1	No 2	5–bis	V	IV	couche
AN	85	70	82	72	59	60	58.5	47
SN (Na nitrate)	–	–	10	–	–	–	–	15
NG	–	6	4	6	11	11	12	10
$BaSO_4$	–	7	4	7	–	–	–	–
$AmmSO_4$	5	9	–	9	–	–	–	–
CC(CollodCott)	10	–	–	–	.05	.05	.05	–
DNT	–	–	–	–	1	2	1	–
NaCl	–	–	–	–	22	22	22	22
Cellulose	–	–	–	–	4.85	4.45	6.45	6.0
Lampblack	–	–	–	–	.1	.5	–	–
Naphthalene	–	–	–	–	2	–	–	–
Carbohydrates	–	8	–	–	–	–	–	–
Dextrin-starch	–	–	–	6	–	–	–	–

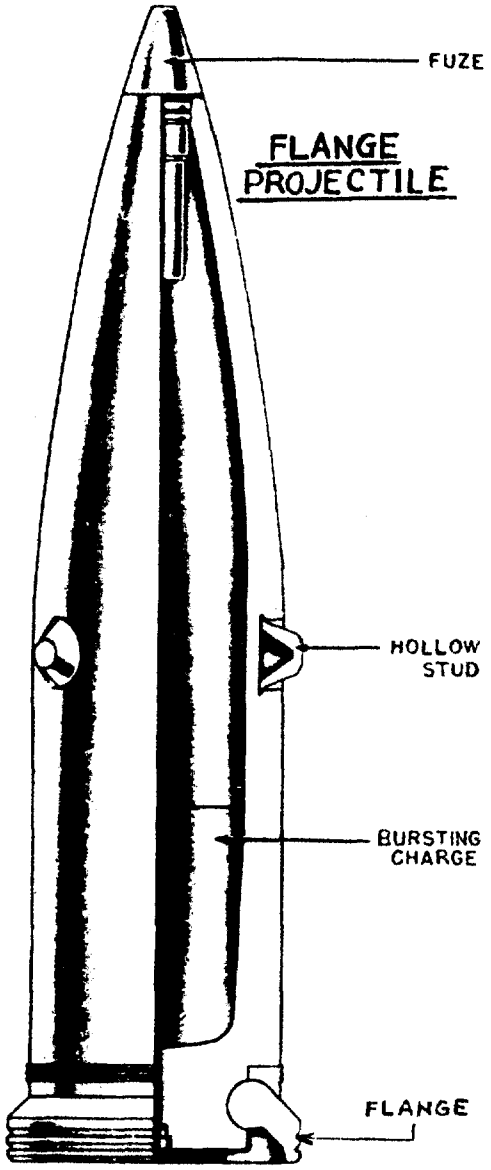


FIG F8

FLARE (Blaze or Glare).

Flare is a pyrotechnic device producing bright, dazzling, broad light intended for signalling, attracting attention, or illuminating the terrain. They may also be used for identification, ignition, location of position or warning

Essentially, a flare consists of a container (metal, cardboard, plastic, paper etc) filled with a pyrotechnic mixture consisting of compressed illuminating composition and an igniting composition (first fire), such as Black Powder or a mixture of Black Powder and the illuminating composition. The first fire composition is usually ignited either electrically by a squib, or mechanically by a spring release pin hitting a primer when a pull wire is withdrawn

Among devices used during WWI, the following may be mentioned:

1) *German White Flare* – contained as illuminating charge, compressed $Ba(NO_3)_2$ 61.5, Al powder 20, sulfur 18.5 (Ref 1, v 1, p 191)

2) *Ground Flares* – These flares, when ignited in a trench or shell hole, were distinctly visible from airplanes even in daytime, but were completely hidden from the enemy. The following compositions were used:

a) $Ba(NO_3)_2$ 82.5, tar 14.5, nitrates of Ca and Mg 3.0%

b) $Ba(NO_3)_2$ 66.4, KNO_3 9.5, powdered Al 7.5, resin (wood tar, pitch) with some oil & wood fiber 15.8 and volatile matter 0.8% (Ref 1, v 1, p 194)

3) *Aircraft Flare* – was invented by the French and consisted of a metal container (cylindrical, bomb-like), loaded with compressed white light illuminating composition [$Ba(NO_3)_2$ 76.00, Al (powdered) 9.75, Al (flaked) 8.25, sulfur (flour) 4.00 and castor oil or vaselin 2.00%] and a first fire composition [$Ba(NO_3)_2$ 44.5, KNO_3 33.3, sulfur (flour) 11.1 and orange shellac (powder) 11.1%]. The tail end of the bomb provided with four fins (to assist the flight) and a large silk parachute. The flare was usually released at a height of about 4000 ft. At about 2500 ft, the parachute was released by means of an expelling charge of Black Powder; following this, the first fire and then the illuminating compositions were ignited. These flares burned with an intensity of not less that 350000 candlepower (Ref 1, v 2, pp 99, 111 & 118 and Ref 3, p 72)

During WWII, much greater varieties of flares were used than in WWI. Most of the flares developed were for airplane use (See Fig 2-75 in Vol 4, p D957)

Following are some flares used by the US Armed Forces:

1) *Aircraft Wing-Tip Flares* – intended for signalling and assisting planes in landing. They were attached to the extremities of the

wings and ignited by "flash igniters", electrically controlled by the pilot. They consisted of a paper tube, 4 inches long and 1¼ inches in diameter, filled with an illuminating composition and closed at both ends with wooden plugs. They usually produced white, red or green lights (Ref 2, p 74)

2) *Flares Released from Aircraft,* such as:

a) *Flare, AN-M26* designed to provide illumination for night bombardments. It was in the shape of a bomb, and was provided with fins and a parachute. The total weight was 52.5 lb. The illuminating composition (which was in compressed form) burned for 3–35 minutes with a yellowish light and a minimum candlepower of 800000. This flare was released at high altitudes at plane speed up to 300 mph (Ref 2, p 315)

b) *Flare, M24* – designed for bombardment, as well as for illumination, weighed 47 lbs and was released from altitudes of from 2500 to 3000 ft at speeds not over 200 mph. In order that the bombardier and plane could be shielded from the glare of the flare, an umbrella-like shade was provided (Ref 2, p 316)

3) *Colored Low-Target Flare, M50* was developed in 1944 when mass concentration of planes for night aerial attacks had grown so heavy that some method of identifying group leaders in the air was required. This flare was towed by a cable behind the plane of each leader, with each leader having a different colored flare. Burning time was five minutes and candlepower 20000 to 225000 (Ref 2, p 316)

4) *Reconnaissance and Landing Flares* were used for illuminating an area at night either to obtain information or for an emergency landing. To this class belonged:

a) *Aircraft Parachute Flare, M9A1.* It was a cylinder 2 inches in diameter and 15 inches long, weighing 2.1 lbs and burning for 1 min with a white light of 60000 candlepower. It was discharged from a special pistol (Ref 2, p 317)

b) *Aircraft Parachute Flare, M8A1* – intended for emergency night landings, weighed 18 lbs, burned for 3 min with a yellowish light of 350000 candlepower (Ref 2, p 318)

5) *Airport Flares* – designed to provide illumination for aircraft landings at emergency fields or recently captured fields before regular electrical illumination could be installed. To these belonged:

a) *Airport Flare, M13* – in the shape of a cylinder, 23 inches long and 1.75 inches in diameter. The composition burned for 3 min at 40000 candlepower

b) *Airport Flare, M76* – larger than the previous one and burned for 5–7 mins at 700000 candlepower

6) *Trip Flares* were constructed like an antipersonnel mine of the *"Bouncing Betty"* type, the fragmentation projectile being replaced by a flare which was projected upwards when anyone came in contact with the device. These flares were intended to give warning of the presence of enemy in their vicinity. The following may be cited as examples:

a) *Trip Flare, M48* – projected a flare, provided with a parachute, to a height of 300 to 500 ft. Time of burning was 20 secs and candlepower 110000 (Ref 2, p 303)

b) *Trip Flare, M49* – designed for the same purpose as the previous one but was not of the bouncing type. It was attached to a pole or tree and functioned by the trip wire being pulled or cut. Time of burning was 60 secs and candlepower 40000 (Ref 2, p 304)

7) *Fusee-Type Flares* were ground flares, designed for troop-recognition purposes or to indicate to cooperating air elements a line of position or direction. A typical flare consisted of a paper cylinder, 7¾ inches long and 1¼ inches in diameter, containing pyrotechnic composition and placed on a wooden block base to which a spike was attached so that the flare could be stuck into the ground. These flares burned for 2 mins; candlepower being 20000 to 35000 (Ref 2, p 306)

8) *Hand Illuminating Flares* could be thrown by hand, like grenades, or launched from a rifle by means of a propellent charge. These flares were the same in design as M49 (See above)

9) *Tree-Suspended Flare* was usually suspended above the ground near the front lines and could be ignited either electrically or by pull wires. In its design, it was a modification of the M26 Flare (Ref 2, p 308)

The following may be mentioned as typical flare compositions used during WWII

a) *White Flare:* $Ba(NO_3)_2$ 66, Al Type B (See Spec JAN-A-289) 26, sulfur 6 and castor oil 2%. A charge of 500g, compressed at 6000 psi, burned for 65 seconds, developing 60000 candlepower

TABLE 1

CHARACTERISTICS OF VARIOUS ILLUMINATING FLARES

Item	*Method of actuation*	*Time lapse from actuation to full function, sec*	*Burning time, sec*	*Candle-power, 10^3*	*Fall, fps*	*Max L, in.*	*Max dia, in.*	*Weight, lb*	*Max speed of airplane at time of release, mph*
FLARE, AIRCRAFT: guide, 1 min									
T6E1 (white)	Electricity	6 to 7	45 to 60	650	.....	5.4	5.46	.....	.....
T7E1 (red)				700					
T8E1 (green)				90					
FLARE, AIRCRAFT: parachute									
M8A1 (w/o suspension bands) (emergency night landing)	Release from airplane	3.0 to 5.0	165 to 195	350	8.0	25.42	4.25	17.6	200
M8A1 (training) (w/o suspension bands)									
M9A1	Fired from PISTOL, pyrotechnic, AN-M8	2.5	60 to 70	60	7.0	15.05	2.0	2.11	200
M26A1 (AN-M26) or M26	Released from airplane	5 to 92	195±15	800	11.6	50.0 (fuzed)	8.0	52.5	150 (M26)
M26A1 (AN-M26) or M26 (w/blue band)				575					350 (M26A1)
M138 (T10E4)		5 to 92	360	1,500	10	45.6	6.25	62	440
M139 (T10E6)			180	3,000					
Mk 5 and Mods		variable	180	600	.....	27.0	4.75	18.0	.....
Mk 6 Mod 5		variable	180	1,000	.....	35.75	5.37	30.0	.....
Mk 6 Mod 6		90							
AN-Mk 8 Mod 1		120	180	500	8.0	25.12	4.75	18	250
AN-Mk 8 Mod 2									
3 minute, electrically operated	.....	1½	180	200	9.1	28	4.5	22	.....
3 minute, Wiley SA 8	.....	.....	.....	.....	.....	.....	.....	.....	.....
FLARE, AIRCRAFT: tow-target, M50		0	360	65	.....	22.8	2.62	7.13	120
FLARE, AIRCRAFT: towed									
Red, M77 (T18)	Tow cable attached to airplane	0	360±30	225	.....	23.34	4.55	21	200
Amber, M78 (T19)				70					
Green, M79 (T20)				90					
FLARE, SURFACE: Airport, M76	Hand or electric squib	0	300 to 420	600 to 850	.....	31.33	4.26	27.6	
Parachute, trip, M48	Pressure or trip wire	3	20	110	3	9.75	5.5	5.0	
Trip, M49	Trip wire	0	55	40	.....	6.75	3.0	1.5	

TABLE 2

HAND-MANIPULATED SIGNALING DEVICES SUMMARY

Item	Launched From	Display	Burn Time (Sec.)	Min. Alt. (Ft.)*	Max. Lgth. (In.)	Max. Diam. (In.)	Approx. Weight (Oz.)
Signal, Illum., Marine, Mk 2 (Very Signal Cartridge)	Pyrotechnic Pistol Mk 5	Free-falling Red, Green, or White Star	6	200	2.43	0.88	1.1
Signal, Smoke, Marine, Mk 2 Mods (Pistol Rocket Signal)	Pyro Pistol Mk 1 or AN-M8	Parachute-Suspended Colored Smoke	20-30	500	14.00	1.68	16.0
Signal, Illum., Aircraft, M11 (Signal, A/C, Red Star, Parachute	Pyrotechnic Pistol AN-M8	Parachute-Suspended Red Star	30	150	7.69	1.57	9.6
Signal, Illum., Aircraft, AN-M37A2 thru AN-M42A2	Pyrotechnic Pistol AN-M8	Two Free-falling Colored Stars (Same or Different Colors)	7	250	3.85	1.57	6.0
Signal, Illum., Aircraft, AN-M43A2 thru AN-M45A2	Pyrotechnic Pistol AN-M8	Free-falling Single Red, Green, or Yellow Star	7	250	3.85	1.57	5.0
Signal, Illum., Aircraft, AN-53A2 thru AN-M58A2	Pyrotechnic Pistol AN-M8	Free-falling Tracer and Two Colored Stars (Same or Different Colors)	Tracer: 2.5 to 4 Stars: 3 to 4.5	250	3.85	1.57	4-6
Signal, Illum., Marine, Mk 1 Mods (Pistol Rocket Signal)	Pyro Pistol Mk 1 or or AN-M8	Mod 0: Free-falling Red, Gr. or Y Star Mod 1: Para-Susp. Red Star	Mod 0:7 Mod 1:20	500	14	1.68	16
Signal, Illum., Ground, M17A1 thru M22A1 (Ground Signal)	Rifle or Carbine Launcher	Odd Nos.: Single Star Colored, Para-Susp. Even Nos.: 5-Star Colored Cluster Free-falling	Star: 20 Cluster: 4	600	10.4	1.9	16-17
Grenade, Rifle, Smoke, M22A2	Rifle Launcher	Green Smoke Cloud on Impact	8	600	10.14	1.88	16
Grenade, Rifle, Smoke, Streamer, M23 and M23A1	Rifle Launcher	Streams of Colored Smoke During Flight	12	600	10.5	1.9	18
Signal, Illum., Ground, M51A1 and M52A1	Rifle Launcher	M51: Para-suspended Single Red Star M52: Free-falling 5-Red Star Cluster	20-30	600	10.5	1.88	16
Signal, Smoke, Ground. M62, M64, and M65	Rifle Launcher	Six Smoke Streamers: M62-Red, M64-Yellow, M65-Green	20	600	10.4	1.88	15
Navy Light, Mk 1 Mods (Fusee, Warning Railroad)	Held in Hand While Functioning	Mod 0 - Red Light Mod 1 - Blue Light	Mod 0: 135 Mod 1: 75	—	9.5	1.45	7
Signal, Smoke and Illum., Marine, Mk 13 (Signal, Distress, Day & Night)	Held in hand while functioning	Day-Orange Smoke Night-Red Flame	20	—	5.18	1.63	6.4

* Above launch point

(Continued)

TABLE 2 *(Continuation)*

Item	Launched From	Display	Burn Time (Sec.)	Min. Alt. (Ft.) *	Max. Lgth. (In.)	Max. Diam. (In.)	Approx. Weight (Oz.)
Signal, Smoke and Illum., Marine, Mk 38	Held in Hand while functioning	Day-Green Smoke Night-Green Flame	Smoke-28 Flame-23	—	5.18	1.63	6.4
Signal, Illum., Ground, M125A1 thru M129A1	Hand Outer case is projector	M125:5 Free-falling Stars. M126-128: Para-susp. colored Star. M129: Para-Suspended Red Smoke	See Para. 5-5-1-8	600	10.14	1.88	21.0
Signal, Illum., Marine, AN-M75	Hand. Outer case is projector	Two Free-falling Red Stars	5	175	5.0	1.25	5.5
Signal, Hand Fired, Mk 80 (Contained in Signal, Kit, Illum., Mk 79, Mod O)	Signal Projector, Mk 31 Mod 0	Single Free-falling Red Star	4.5	250	2.25	0.52	12
Signal, Illum., Aircraft, Mk 6 Mod O (A/C Emerg. Ident. Signal)	Hand-thrown From Aircraft. Grenade Type	Para-Suspended Red, Green, or White Star	25	—	5.95	2.6	18
Signal, Smoke, Aircraft, Mk 7 Mod O (A/C Emerg. Ident. Signal)	Hand-thrown From Aircraft. Grenade Type	Para-Suspended Red, Green, Yellow, or Black Smoke	25	—	9.57	2.5	28
Fusee, Warning, Railroad, M72	Ground (Hand-Emplaced)	Red Flame	20 Minutes	—	15.88	0.9	10
Grenade, Hand, Smoke, HC, AN-M8	Hand-Thrown	White Smoke Cloud	105	—	4.5	2.5	23
Grenade, Hand, Smoke, M18	Hand-Thrown	Red, Green, Yellow, or Violet Smoke	50	—	4.5	2.5	23
Cartridge, Signal, Mk 130	38-Caliber Revolver	Red Streak	5.5 - 7.5	800	1.55	0.379	0.39

* Above launch point

b) *Yellow Tinted Flare:* $Ba(NO_3)_2$ 34, Magnesium Type III (Spec JAN-M-382A) precoated with 6% linseed oil 36, Al Type B (See Spec JAN-A-289) 8, Na oxalate 20, linseed oil 1 and castor oil 1%. Chge of 6300g, compressed at 3600psi burned for 280 secs, developing 556000 candlepower

Modern Military Flares. Today flares are used primarily for illuminating and signaling. Characteristics of various illuminating flares are shown in Table 1 (Ref K) and those of signaling flares are shown in Table 2 (Ref M). Illuminating flares produce essentially white light, have an intensity in foot-candles adequate to produce a brightness level from 0.1 to 1.0 foot-lambert and burn at peak intensity for a minimum of 30 secs. Signal flares are smaller and faster burning than illuminating flares. They consist of one or more colored stars with or without colored tracers. They are usually supported by a parachute and are fired from a flare pistol or projector. Aircraft parachute flare, MK5, a typical illuminating flare is shown in Fig 1, while a typical tow target illuminating flare is shown in Fig 2. Two typical signaling flares are shown in Figs 3 & 4

Refs: 1) Faber, Pyrotechnics (1919), Vols 1 & 2 2) Ohart (1946), 8, 269, 303–08 & 315–18 3) Weingart, Pyrotechnics (1947), 72–4 4) Anon, "Military Pyrotechnics", Dept of the Army Tech Manual **TM 9-1370-200** (Sept 1966), Chapter 3, "Flares", pp 3-1 to 3-65

Addnl Refs: A) W.F. Shirk, "MK III Reconnaissance and Observation Flare", PATR **1130** (1941) B) C.R. Hoover, "Development of Flares Suitable for Underwater Use", OSRD **435** (PBL 40558) (1942) C) A.

Stettbacher, Nitrocellulose **13**, 203–7, 224–9 (1942) & CA **37**, 5240 (1943) D) G.A. Hill & R.G. Clarke, "Underwater Flares", OSRD **1522** (PBL 24890) (1943) E) British Report, "Comprehensive List of Govt Explosives 1955, Admiralty **BR 819** (1B/54) F) J.F. Tyroler & L.J. Frey Jr, "Development of a Special Flare (Rita)", PATR **2238** (1956) G) Anon, "Flares for Battlefield Illumination", Proj NR AVN **1857**, Dept of Army, Aviation Board (May 1958) H) "Flares", Special Index, V 2, 1 Oct 1958, pp 90–1 I) T.H. Johnson et al, "Flare-Device", USP 2868129 (1959) J) C.A. Knapp, "New Infrared Flare Compositions (U)," **Proj 504-01-031**, Dept of the Army (July 1963) J_1) D.E. Middebrooks, S.M. Kaye & G. Weingarten, "The Effects of Processing on Pyrotechnic Compositions. Part 3. Dimensional Effects of Paper Cases on Illuminants and Burning Rate of Flare Compositions", PATR **3275** (Jan 1966) K) Anon, "Engineering Design Handbook, Military Pyrotechnics Series", Part One, *"Theory and Application"*, **AMCP 706-185** (1967) L) P.L. Blackshear, Jr et al, "On the Quality and Performance of Flares", 12th SympCombstn Paper **128** (1968) M) Anon, Pyrotechnic, Screening and Dye Marking Devices, **NAVORD OP 2213** (1968)

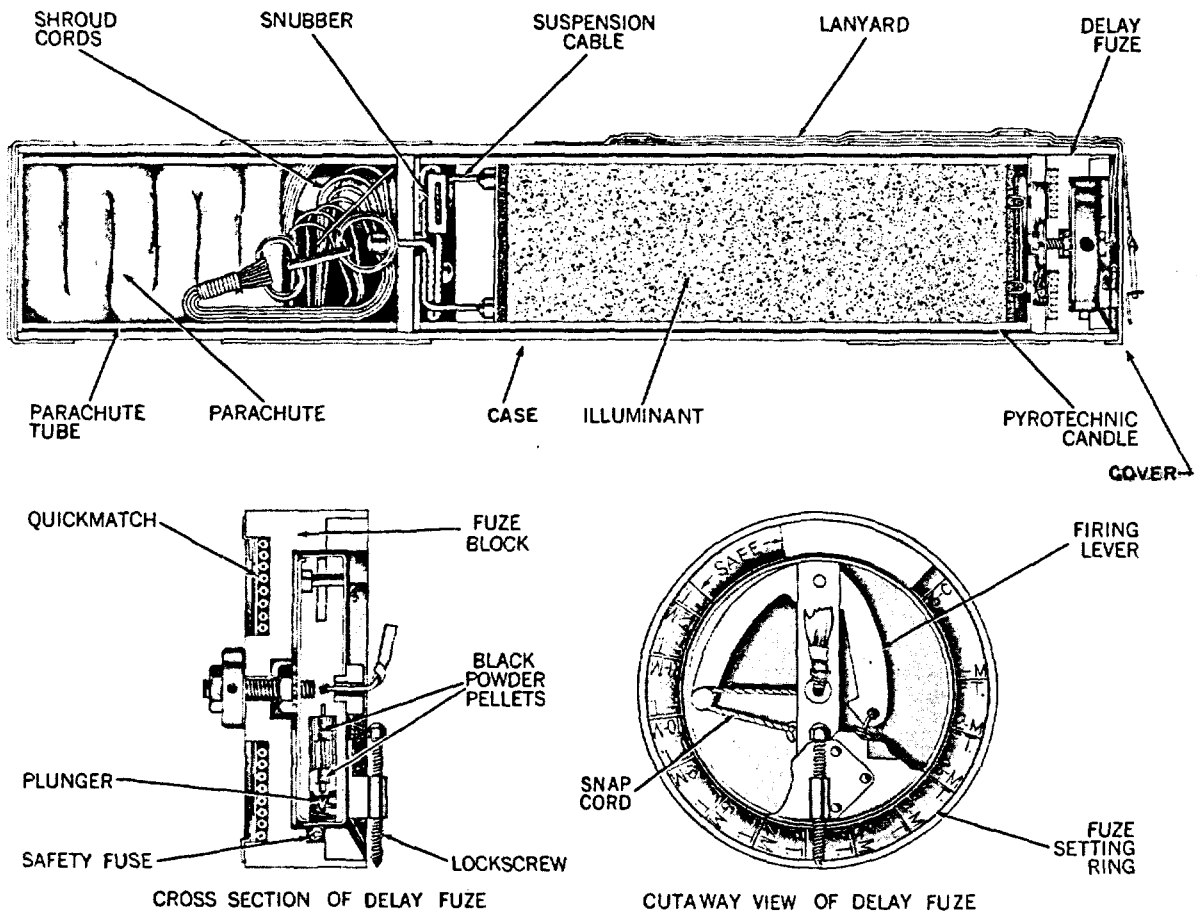
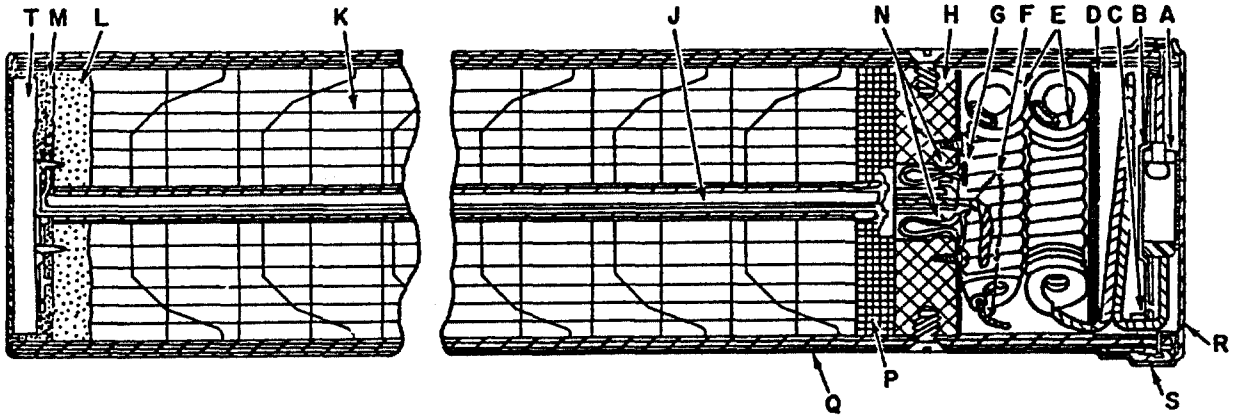


FIG 1 Aircraft Parachute Flare, Mk 5



A- CABLE RING
B- PULL COVER
C- SLEEVE
D- SAFETY DISK
E- SHOCK ABSORBERS
F- FRICTION WIRE PULL WIRES
G- RETAINER
H- BASE BLOCK
J- QUICKMATCH
K- ILLUMINANT CHARGE
L- FIRST-FIRE CHARGE
M- PRIMING COMPOSITION
N- IGNITERS
P- FIRE CLAY TOP SEAL
Q- FLARE CASE
R- CLOSING COVER (TOP)
S- SEALING STRIP
T- CLOSING COVER (BOTTOM)

FIG 2 *Typical Aircraft Tow Target Flare*

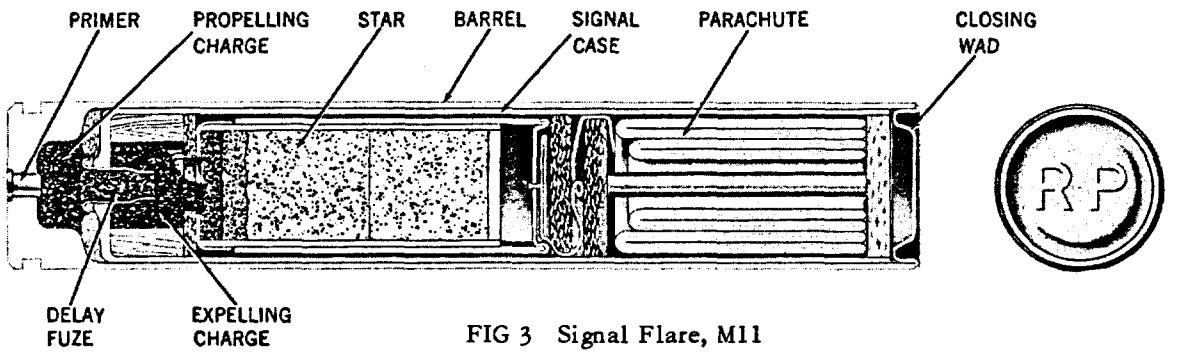


FIG 3 Signal Flare, M11

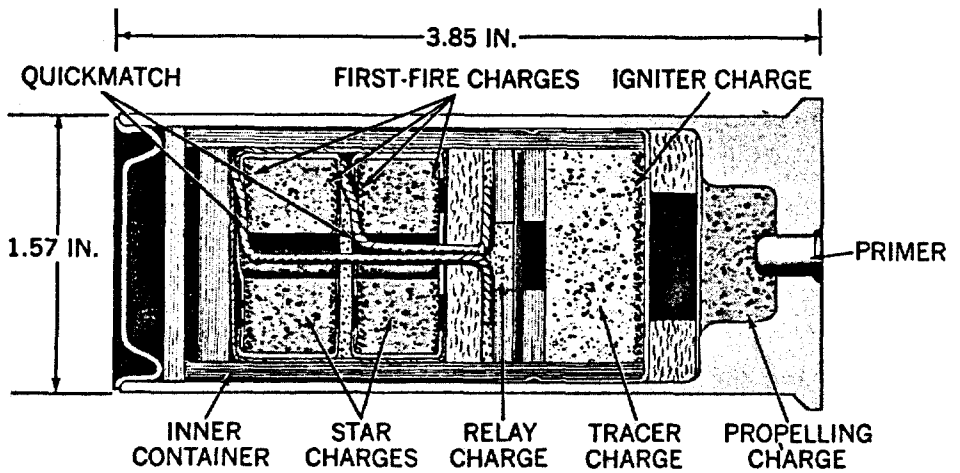


FIG 4 Signal Flare, M53A2–M58A2

Flare, Aircraft. See under FLARE

Flare, Airport. See under FLARE

Flareback or Backfire. See under Backflash in Vol 2 of Encycl, pp B2-R to B3-L

Flare, Float and Flare, Surface. A *float flare* is a pyrotechnic signal launched from an aircraft to mark a location at sea. It floats on the surface and emits smoke and flare for up to one hour. A *surface flare* is a pyrotechnic item for use in surface position, ground or water, designed to produce a single source of intense light for purposes such as illumination of airport runway and warning of infiltrating enemy troops
Ref: Glossary of Ordn (1959), 119-L & R

Flare-Fusee. See under FLARE

Flare, German. (Leuchtkugel oder Fackel). These flares usually consisted of a cylindrical container housing an illuminating element. Upon being ignited by a pull friction igniter or a time fuze, the flare burned vigorously producing intense light and heat. The illuminating element consisted either of a single or a multiple candle unit which varied in intensity of illumination and color. Flares were made with or without parachutes

A brief description of the following flares was given in Refs 1 & 2, and they are illustrated in Fig F9

1) LC 10 (Leuchtcylindrisch 10) consisted of an Al cylinder, a single candle in a cardboard liner, an "89" clockwork fuze and a parachute located in the tail end. The flare was dropped from a plane and at a predetermined time the fuze fired and ejected the candle and its parachute from the body. Simultaneously the candle was ignited

2) FB 50, Single Candle Parachute Flare Flare is described on p 66 of Ref 1

3) LC 50F Ausf C Parachute Flare consisted of a cylindrical Al body with dome-shaped nose attached by means of brass screws. On releasing the flare, the pyrotechnic delay (inside the fuze) was ignited. This fired the quickmatch, which in turn burned thru the flash tube and ignited the Black Powder charge in the tail. The pressure of the gases developed by the deflagrating BkPdr caused all four flare candles and the parachute to be expelled thru the nose, after shearing the holding screws. Simultaneously, the candles were ignited thru perforations in the ejector plate. The compn of the candle was Ba nitrate 75.8, Al 16.5 and S 7.7%. The burning time was slightly over 5 min and the candlepower 216000

4) LC 50F Ausf E, Single Candle Parachute Flare is described on pp 68–9 of Ref 1

5) *LC 50F Ausf G, Single Candle Parachute Flare* is described on pp 69–70 of Ref 1

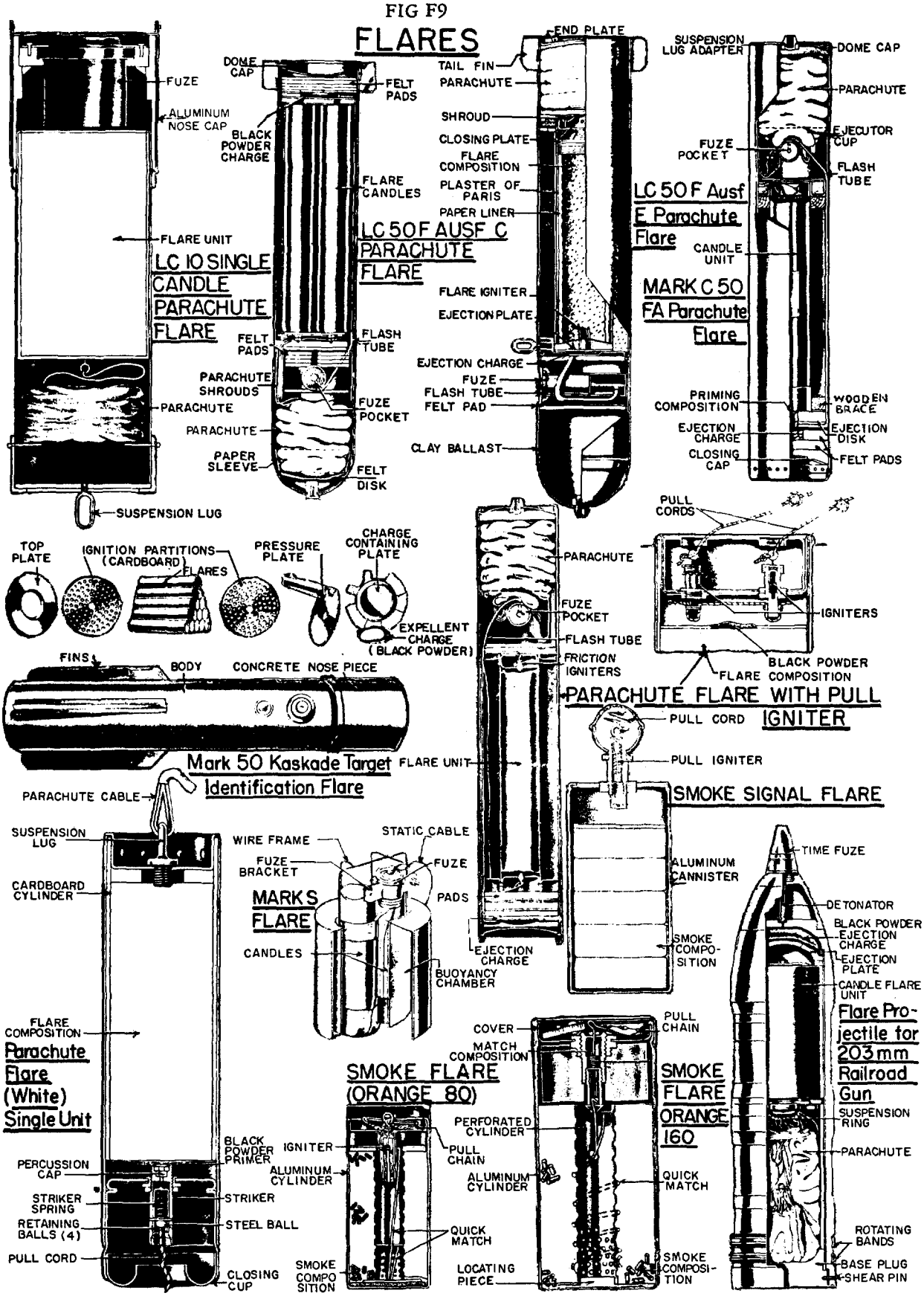
6) Mark C 50 F/A Parachute Flare consisted of a cylindrical Al housing contg a parachute, fuze, quickmatch, single candle unit, flash tube, priming compn and ejection disk. When the flare was released, the aerial burst fuze started to function. The flash ignited the quickmatch and the flame was transmitted thru the flash tube to the tail end to ignite the ejection disk of BkPdr. The pressure of the gases developed by the burning BkPdr expelled the parachute and the candle thru the nose. Simultaneously the primer compn and the candle were ignited

7) Mark 50 Kaskade Target Indicating Flare consisted of a cylindrical sheet metal container 7.7 inches diam and 41.0 inches long contg 62 flares (in three layers separated by perforated cardboard partitions), an expelling charge of BkPdr, smokeless propInt ignition disks and an igniter (fuze) assembly. A heavy concrete nose was provided to make the missile fall with the nose downwards, when released from a plane. As the missile fell, the expelling charge was ignited, thus ejecting the flares (candles). At the same time the propInt ignition disks ignited each candle. (Composition of candles is given under Pyrotechnics [See also BIOS Rept 1233 (1946), p 1]

8) Single Candle Parachute Flare with Pull Igniter was similar in construction to the Mark C50F/A flare. The principal difference

FIG F9

FLARES



was that the candle was reversed and ignited by pull (friction) igniters instead of by a BkPdr charge. After the flare was released from the aircraft, the fuze (thru the flash tube) ignited the ejection charge of BkPdr and the pressure of the gases ejected the parachute and the candle thru the nose. At the same time the parachute pulled the cords of the igniters, which were provided with delay elements of 3½ secs. The candle was then ignited and burned for 5 minutes

9) Single Candle Unit Parachute Flare (White) consisted of a cylindrical Al body which was attached to a parachute by means of a cable. Eight shroud lines terminated in a loop which was in turn attached to the pull cord of the igniter. On releasing the flare, the parachute exerted a pull on the igniter "31" firing cord thus releasing the striker spring. Then the striker hit the percussion cap igniting the BkPdr primer and the candle

10) Single Candle Parachute Flares: I (White) and *II (Red)* are described on pp 75–7 of Ref 1

11) Mark 5 Flares, Types 1 and 2 consisted of a cylindrical buoyancy chamber which contd two candles. To these were attached a fuze, a static cord and a pull igniter. The static cord functioned either the arming device of the fuze or the pull igniter. When the device was released (from a container) over the water it went under the surface and then came up. It floated with the head of the flare just clear of the water. When the 1st candle was about ¾ burned out, a piece of safety fuse running to the 2nd candle was ignited and, after a short delay, the 2nd candle started to burn. Each candle burned for about 2¾ min

12) Smoke Flares: Orange 160 and Orange 80, used as wind drift indicators, are described on pp 79–80 of Ref 1

13) Smoke Signal Flare, used as navigation aids by pilots, is described on p 80 of Ref 1

14) Smoke Signal Flare, ARDR, used for the same purpose as above, is described on p 86 of Ref 1

15) Distress Signal Torch consisted of a narrow sheet Al cylinder contg three pressed charges of flare compn which burned respectively red, white and red. The compns were ignited by a pull igniter

16) Ground Flare, Bodenleuchte (P) Fi56 217 was briefly described in BIOS Final Report 1233 (1946), p 2 and the compn of the flare was given under Pyrotechnics

In addn to flares dropped from planes, there were some flares fired from guns, eg the Flare Projectile for the 203mm Railway Gun *(20.3cm Leuchtgranate)* described in TM 9–1985–3 (1953), pp 519–20. The shell was conventional in design except that it had an additional bourrelet machined near the middle of the shell body. The weight of the shell was 226¼ lbs, that of the flare candle unit and parachute assembly 47 lbs, and of the expelling charge (BkPdr) ½ lb. The flare and parachute were expelled thru the base of the shell

Refs: 1) Anon, TM 9–1985–2 (1953), pp 65–81 2) Fedoroff et al, PATR **2510** (1958), pp Ger 49–50

Flare, Ground. See under FLARE

Flare, Guide. An electrically ignited *aircraft flare* for attachment to an aerial bomb, which produces a very bright light, either white or colored, to mark the position of the bomb and permits its guidance to the target

Ref: Glossary of Ordn (1959), 119-L

Flare, Guided Missile. A pyrotechnic item designed to produce a single source of intense light for the purpose of visually tracking a guided missile during its flight to a target (Excludes "Guided Missile Tracer" which provides a signal to permit tracking of missile)

Ref: Glossary of Ordn (1959), p 119-L & 297-R (Tracer, Guided Missile)

Flare, Hand Illuminating. See under FLARE

Flare, Illumination. A flare designed to provide a constant, uniform illumination level on the ground was patented in 1973 (Ref 1). Current flares burn with a constant candlepower output and when they descend, the illu-

mination on the ground – proportional to the square of the distance from the ground – continually increases. The new flare has successive increments, each burning with reduced output. The changes in output are achieved by varying the particle size of the metallic fuel. For example, when using Mg/NaOH/Laminac flare mix, particle size of Mg is 125, 200 & 350 microns for three increments, respectively
Refs: 1) J.F. Tyroler, USP 3744418, July 10, 1973 2) G. Cohn, Expls&Pyrots **7**(2), 1974

Flare, Landing. See Reconnaissance and Landing Flare, under FLARE

Flare, Long Burning. A make-shift device, invented on the Korean front by C.J. Husch, was constructed from two empty shell cases filled with a mixture of Diesel fuel and gasoline and placed with an ordinary trip flare in a mortar container, previously filled to a certain level with Napalm, or jellied gasoline. When the flare was ignited, it set fire to the Napalm and heated the fuel (in shell cases) to the boiling point. The ensuing pressure forced burning vapor 6 to 10 feet into the air thru the perforations in the tops of the cases, and the brilliant yellow flame resulted, which lighted up surrounding terrain for more than 5 hours
Ref: Anon, Ordnance **36**, No 190, 620(1952)

Flare, Magnesium. A general term indicating a flare using Mg as the illuminating agent
Ref: Glossary of Ordn (1959), 119-L

Flare, Parachute. A pyrotechnic device attached to a parachute and designed to provide intense illumination for a short time. May be discharged from aircraft or from the surface
Ref: Glossary of Ordn (1959), 119-L

Flare, Parachute, Hand Fired. A complete self-contained device which is fired from the hand, and which provides a parachute borne pyrotechnic light
Ref: Glossary of Ordn (1959), 119-L

Flare Pistol, Pyrotechnic Pistol or Very Pistol. A single shot device designed specifically for projecting pyrotechnic signals. This item may or may not be provided with a method of mounting to an adapter
Ref: Glossary of Ordn (1959), 213-R (Pistols, Pyrotechnic)

Flares, Pyrotechnic for Cloud Seeding. Electrically or percussion-initiated flares, called "nuclei generators", are being used to dispense nucleating silver iodide and salt particles into clouds as a means of altering precipitation or controlling hailstorms, fog, lightning, violent hurricanes and tornadoes. Custom-made flares range from 2 to 18 inches in length; longer ones have 1½ inch diameter. By controlling the chemical properties of the pyrotechnic mix, maker controls composition, burning rate and temp of combustion. Burning times range from under 45 seconds to 20 minutes. One item produces up to 25 grams of effective nuclei per minute for 4½ minutes. A single gram of silver iodide can form billions of small ice crystals when sprayed into a cloud holding supercooled water droplets. Such crystals grow and fall of their own weight as snow or rain, depending on temps below the nucleating area. Clouds that harbor large water droplets are fertile for seeding: droplets form from molecules when the volume of molecules exceeds a certain critical radius, and when enough of them collide simultaneously. Maker's first field trial seeded warm clouds with salt nuclei to disperse fog; experiment increased visibility by as much as one mile. Flares can be fired from aircraft wing racks, or a standard Very pistol.
Refs: 1) Olin-Mathieson Chemical Corp, 460 Park Ave, New York, NY 10022
2) Expls&Pyrots **1**(6)(1968)

Flare, Reconnaissance. A former term for an aircraft flare used in air reconnaissance to light up the ground
Ref: Glossary of Ordn (1959), 119-L & R

Flare, Surface. See under Flare, Float and Flare, Surface

Flare System, Pyrotechnic. Experimentation with an advanced pyrotechnic flare system at Picatinny Arsenal, as reported in a technical paper at the 1970 Army Science Conference, indicates it offers several advantages over solid flares in current use. The system uses gas and thus has the advantage of on-off control, in contrast to solid flares which, once ignited, have to burn out or be extinguished and cannot be used again. Using a mixture of gases to produce bright white flames, the system permits illumination levels and burning times to be varied readily, and it has a time range of a fraction of a second to hours. Most solid flares burn only a short time

While solid flares are smoky and therefore subject to wind and other weather conditions, the pyrotechnic system is relatively free of particulate matter and can be made directional. In testing, directional light efficiencies have been increased 12 times by using an aluminum reflector to produce efficiencies of one-quarter to one-half million candleseconds per gram

Another advantage of gaseous systems is that they do not require atmospheric oxygen. The reproducible, homogeneous mixtures can be used and stored for long periods at high and low temperatures without loss of efficiency. Gas systems, which can be used on the ground or dropped by a parachute, require a burner, two tanks and an emitter. They can be ignited by remote control, either by electric fuse or an electric spark
Refs: 1) Army Res & Dev News Magazine **11**(5)(1970) 2) Expls&Pyrots **3**(12)(1970)

Flare, Tow-Target. See Colored Tow-Target Flare, M50, under FLARE

Flare, Tree-Suspended. See under FLARE

Flares, Trip. See under FLARE

Flash. A sudden, transitory outburst of flame or light, which may also be defined as a sudden luminous temporary flame
Refs: 1) Hackh's Dict (1946), 344-R
2) "The American College Dictionary", Random House, NY (1952), 460-L

Flash. NOLTR **1111** (1952), p G2 refers to the method of initiating an explosive loaded element by heat or flame from another element

Flash-Across, Heat-Pulse and Hypervelocity Phenomena, in Detonation. See Vol **4**, pp D348-R & D349-L

Flash and Flame. When Lead Azide was shot in a non-expl atmosphere, an intense luminosity was produced. This light emission was greatest in gas of low molecular specific heat, where the latter does not increase with temperature. The luminosity decreases the greater the specific heat and the temperature coefficient. The following gases were examined and found to have this decreasing order of luminosity: $N_2 + H_2$, CO_2, acetylene & butane. The effect was also observed to increase with the density of the surrounding gas, eg, H_2, N_2, O_2, Cl_2 or He, Ne, A & Kr. The luminosity was described as very feeble in H_2 or He. The expln of 0.4ml of a 3:1 mixture of TNM-toluene in argon gave a flash lasting under 5 microsecs with a brilliancy of 2.5–10 million candles. The effect was attributed to heating of the gas by adiabatic compression by the expln wave and to the dissociation of molecules and

ionization of atoms. The increase in the series He, Ne, A, Kr followed the order of decreasing ionization potential. The formation of atomic oxygen and of ionized gases can be a factor in safety of expls in gassy coal mines
See Vol **3**, p C448-R, Problems 1 and 2
Refs: 1) A. Michel-Lévy & H. Muraour, CR **198**, 2091–3 (1934) & CA **28**, 4907 (1934) 2) Ibid, **200**, 543–5 (1935) & CA **29**, 2451 (1935) 3) Ibid, **200**, 924–6 (1935) & CA **29**, 3160 (1935)

Flashback. Same as Backflash or Flareback described in Vol **2**, pp B2-R & B3-L

Flashback Fuze. See Fuze, Spitback

Flashback (or Spitback) Tube. A tube attached to the truncated apex of a shaped charge liner, ordinarily extending thru the the explosive charge. Thru this tube the detonating impulse is transmitted from the point of initiation to a detonator at the base of the explosive charge
Ref: Glossary of Ordn (1959), 270-R (Spitback Tube)

Flashbomb. See Photoflash Bomb, under Bombs, Pyrotechnic in Vol **2**, p B229-L

Flash Charge. A readily ignitable explosive charge used in ignition elements of electric primers and detonators. Its function is usually to ignite a subsequent charge of lesser sensitivity and greater brisance
Ref: Glossary of Ord (1959), p 65-L (Charge, Flash)

Flash Charge of Electric Squib. Its compn is DADNPh (JAN-D-552) 20±2%, $KClO_3$ (MIL-P-150C Grade I, Class C) 60±5%, powdered wood charcoal (JAN-C-178A) 15 2%, Nitrostarch (N 12.75% min) 5±0.5%. Another compn is DADNPh 19%, $KClO_3$ 66%, powdered wood charcoal 13%, Nitrostarch (N 12.75%) 2% (moisture 0.3% max). The 2nd of these mixts was analyzed at Picatinny Arsenal by the following methods:
Moisture. Transfer an accurately weighed portion of approx 1 g of the flash charge compn to a tared moisture dish 2 inches in diameter. Place the moisture dish and contents in a drying oven at 80°–85°C for 2 hours, cool in a desiccator and weigh. Calculate the loss in weight of the dish and contents to percent moisture in the sample

Percentage of moisture = 100A / W

where: A = loss in wt of moisture dish & contents in grams
W = wt of sample in grams on a dry basis

Diazodinitrophenol (DADNPh). Transfer an accurately weighed portion of approx 1 g of the sample to a 250ml beaker and add 150ml of ethylene chloride. Heat the beaker and contents on a steam bath for 2 hrs with occasional stirring. Filter the contents of the beaker with the aid of a tared medium-sized porous-glass crucible and suction. Wash the beaker and residue in the crucible with boiling ethylene chloride until the filtrate coming thru the crucible is no longer colored yellow. Dry the crucible and residue in an oven maintained at 80°±5°C for 2 hours, cool in a desiccator and weigh. Calculate the percentage of Diazodinitrophenol in the sample on a dry basis as follows:

$$\text{Percentage of DADNPh} = \frac{(A - B)}{WC} 100$$

where: A = wt of glass crucible and sample in grams
B = wt of glass crucible and residue in grams
W = wt of sample in grams on a dry basis
C = percent solubility of DADNPh in ethylene chloride obtd as follows:
Place 0.1900±.0010g of the dry Diazodinitrophenol from the lot used in the manufacture of the flash charge composition being analyzed in a 250ml beaker and add 150ml of ethylene chloride. Heat the beaker and contents on a steam bath for 2 hours with occasional

stirring. Filter the contents of the beaker with the aid of a tared medium-sized porous-glass crucible. Wash the beaker and residue in the crucible with hot ethylene chloride until the filtrate coming thru the crucible is no longer yellow in color. Dry the crucible and residue in an oven maintained at 100°±5°C for 1 hr, cool in a desiccator and weigh. Calculate the loss in weight of the crucible and contents to percent solubility of DADNPh in ethylene chloride. This value is represented by the letter "C" given in the equation for calculating the percentage of DADNPh. This solubility was found to be 97.26% as determined on a batch of DADNPh. Use this value of 97.26% except in case of non-compliance, dispute or question[a]. In such cases use the value determined as described above

Remark:

a. In connection with developing a method for the detn of the DADNPh content, it was found that DADNPh was not completely soluble in ethylene chloride solution and that this insolubility adversely affected the accuracy of the DADNPh detn. Therefore, it was considered advisable, in the method for the detn of the DADNPh content of the flash chge compn, to prescribe a correction equivalent to the solubility of the DADNPh. This solubility was found to be 97.26% when detd using one lot of DADNPh purchased from Hercules Powder Co. It is recognized that the solubility value of 97.26% may be significantly different for different lots of DADNPh. However, because of the non-availability of a sufficient number of different lots of DADNPh used in the flash chge compn, no addnl work was done to establish the reliability of the solubility value of 97.26% or to determine whether a solubility value could be established which is invariant of the different lots of DADNPh used. In this connection it is intended that, in the course of the analysis of samples of flash chge compn, the solubility will be detd and recorded, whenever practicable, using various lots of DADNPh

Nitrostarch. Extract the dry residue obtained in the above determination with ten 5ml portions of hot water, stirring the residue in the crucible after the addition of each portion before applying suction. Dry the crucible and residue obtained after the water extraction of the potassium chlorate with ten 5ml portions of hot acetone, stirring the residue in the crucible after the addition of each portion before applying suction. Dry the crucible and residue in an oven maintained at 100°±5°C for 2 hours, cool in a desiccator and weigh. Calculate the percentage of Nitrostarch in the above sample on a dry basis as follows:

$$\text{Percentage of NS} = \frac{100\,(A - B)}{W}$$

where: A = wt of glass crucible & residue before extraction with acetone, in grams

B = wt of glass crucible & residue after extraction with acetone, in grams

W = wt of sample in grams on a dry basis

Powdered Wood Charcoal. From the wt of the glass crucible and residue after extraction with acetone and the wt of the glass crucible calculate the percentage of powdered wood charcoal on a dry basis as follows:

$$\text{Percentage of wood charcoal} = \frac{100\,(A - B)}{W}$$

where: A = wt of glass crucible & residue after extraction with acetone, in grams

B = wt of glass crucible in grams

W = wt of sample in grams on a dry basis

Potassium Chlorate. Calculate the percentage of potassium chlorate in the sample on a dry basis by subtracting from 100 the combined percentages of DADNPh, Nitrostarch and powdered wood charcoal

Ref: J. Campisi, "Development of Methods of Analysis of the Flash Charge for Electric Squib", Report from the Chemical Laboratory of Picatinny Arsenal, Dover, NJ, Rept No **126836**, 14 July, 1949

Flash Composition-Lined Smokeless Propellant-Loaded Cartridge Shells. Shell shaped like shotgun shell, lined with a flash compn (oxidizing agent and aromatic nitro compd) loaded in grain colloided smokeless proplnt (grain 0.240 inch dia. x 0.260 inch long). Flash compn causes uniform ignition suitable for starter for

internal combustion engines
Ref: A.M. Cohan (Atlas Powder Co), USP 2429528 (1947) & CA **42**, 764 (1948)

Flash Depressor. A substance used to reduce the flash from a rocket motor
Ref: Glossary of Ordn (1959), 119-R

Flash Distillation. Distillation in which an appreciable proportion of a liquid is converted to vapor in such a way that the final vapor is in equilibrium with the final liquid
Ref: CondChemDict (1961), 499-R

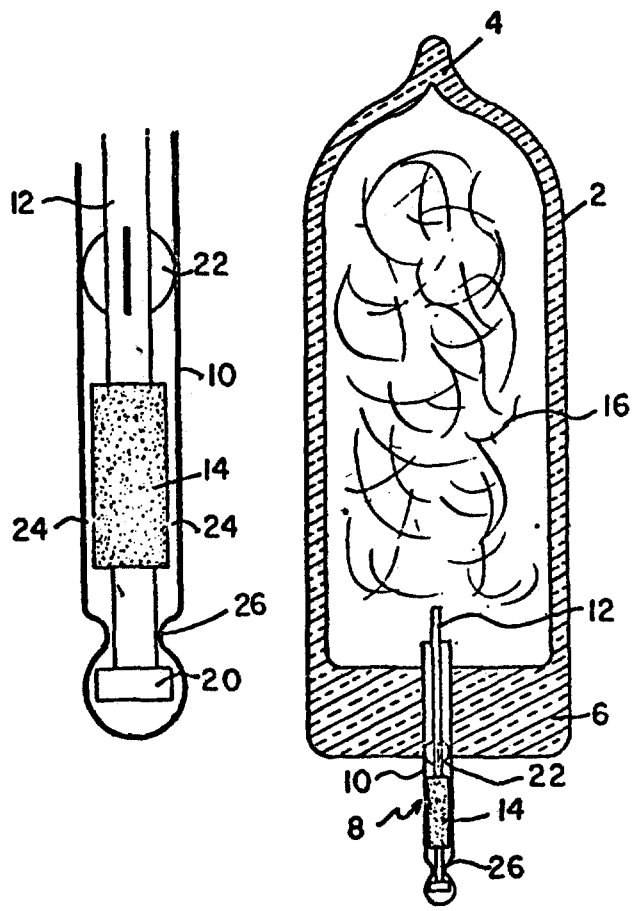
Flash Fuse. A small explosive device similar in appearance to a detonator, but loaded with LE (low explosive), so that its output is primarily heat (flash). Usually electrically initiated, and employed to initiate action of pyrotechnic devices and rocket propellants. It is one of the *squibs.*
Ref: Glossary of Ordn (1959), 272-R (under Squib)

Flash Hider or *Muzzle Flash Suppressor* (Dämpfer, in Ger). A metallic cone and/or flat disks which are attached to the muzzle of a gun to conceal the flash when the gun is fired and to prevent temporary blindness of the crew while firing
Refs: 1) Glossary of Ordn (1959), p 119-R
2) Anon, "Ordnance Design Handbook, Elements of Armament Engineering", ORDP **20-106** (Aug 1960), p 3-21

Flash Photography Without Battery. In order to overcome the failure problems of contacts and batteries in ordinary flash cubes, Sylvania Co invented before 1970 a flashcube, called "Magicube" which is percussion activated. Its reliability is estimated at 99.7%. The flashcube works as follows:

An initiating composition is coated on a wire centered in the tube, the wire acting as an anvil for this percussion initiating action. The mechanical blow is provided by a torsion spring in the form of a hair pin that is triggered by a pin in the camera. When the picture is taken, the shutter button also pushes up the flashcube pin. The pin lifts the torsion spring from its latch so that the free spring end (the striker) strikes the outside of the metal tube to squeeze the initiating compn between deformed tube and wire anvil. Thus ignited the initiating compn flashes the light-producing zirconium mix in the lamp. Less than 0.2 inch-ounces of energy is required to lift the striker from the latch. It takes 0.3 milliseconds for the striker to travel from latch to tube. The striker develops energy from its spring action in excess of 3 inch-ounces. The cube consists of four lamps, each having its own spring striker

The accompanying drawing is taken from the basic US patent 3535063. Pertinent call-outs are: (2) lamp envelope, (8) initiating assembly, (10) metal tube, (12) wire anvil,



(14) initiating compn, (22) protuberances designed to center the wire in the tube and to prevent the tube from touching the initiating compn, (26) crimp in tube to anchor the wire. Several initiating compns were patented, two are as follows:

Patent 3540818

Boron	21.17%
Zirconium	19.53
Stabilized red phosphorus	28.63
Sulfur	0.03
Potassium chlorate	28.63
Magnesium oxide	0.27
Hydroxyethyl cellulose	1.74

Patent 3674411

Titanium powder	48.01%
Potassium chlorate	24.85
Red phosphorus	24.85
Hydroxyethylcellulose	1.30
Magnesium oxide	0.66
Sodium lignin sulfonate	0.23
Sodium 2-ethyl-hexylsulfate	0.03
Trichlorophenol	0.04
Sulfur	0.03

The basic construction has been modified in addnl patents. One change added a refractory bead to the top of the wire (inside the lamp) to prevent burn thru and to disperse the hot particles of initiating material. Addnl patents covering the initiation of the Magicube are: 3535064, 3540819, 3540820, 3597603, 3597604, 3602618, 3625641, 3667992, 3699021, 3700377, 3730669, 3734679

Refs: Public Affairs Dept, Sylvania Electric Products Inc, 730 Third Ave, New York NY 10017 2) G. Cohn, Expls&Pyrots **6**(12), 1973

Flashes, Ignition and Propagation of. Flash radiographs taken by Hershkowitz (Ref) after initiation of a small quantity of expl centrally located in a case contg oxidant/metal (K perchlorate/atomized Al) compn show an expanding bubble of gaseous products, surrounded by an expanding spherical shell of compressed compn. High-speed framing camera photographs of the test item indicate ignition occurs at the outside periphery of the spherical shell of compn as a result of impact of this shell with the case and by flow of thermal energy during the period of case deformation

The interrelation of expl, compn, and case parameters in determining oxidant/metal flash performance are discussed qualitatively. Steps necessary to achieve a quantitative theory are outlined

Ref: J. Hershkowitz, "The Mechanism of Ignition and Propagation of Oxidant-Metal Flashes", PATR **2526** (April 1958)

Flashing Bullets. Bullets, producing flash of light on impact with a target to indicate the point hit, are charged with a compn contg Al and $KClO_3$, as for instance: Al 40, $KClO_3$ 30, TNT 20, $Sr(NO_3)_2$ 6 & glass 4%

Refs: 1) W.H. Buell, BritP 20307 (1914) & CA **10**, 694(1916) 2) W.H. Buell, USP 1242879(1917) 3) W.H. Buell, USP 1242900 (1917) & CA **12**, 225(1918) 4) P.P. Alexander, USP 2611316(1952) & CA **47**, 5686(1953)

Flashing of Explosive Substances. The flashing characteristics of expl substances is not detd by the relation betw the bp and flash point. On rapid heating to high temp some expls show flameless decompn instead of a flash because of the small concn of the decompn products at the bp. It is these decompn products which are responsible for self-inflammation. Vapors of expls which have a much higher temp of self-inflammation than the bp, decompose without a flash; however at a higher temp, self-inflammation takes place again. Values for bp at 2mm (exptl), 50mm (exptl) and 760mm (most probable value), ignition temp and latent heat of vaporization (cal/mol) for several expls are: $MeNO_3$ 5°, 66°, ——, 4000; $C_2H_4(NO_3)_2$ 70°, 125°, 197±3°, 195–200°, 6500; TNT 190°, 245–250°, 300±10°, 295–300°, 10700; PA (Picric Acid) 195°, 255°, 325±10°, 300–310°, 6900; TNB (Trinitrobenzene) 175°, 250°, 315±10°, ——, 7600; PETN 160°, 180°, 200±10°, 215°, 17300; Nitroglycerin 125°, 180°, 245±5°, 200°, 7100. The ignition temp approx coincides with the bp at 760mm which indicates that inflammation is preceded by the formation of a large amt of vapor which ignites when heated. TNT, PETN or PA neither detonate nor burn in vacuo, probably because the bp in vacuo is not near the ignition temp

Refs: 1) A. Belyaev & N. Yuzefovich, DoklAkadNauk **27**, 133–5 (1940) (in English) & CA **34**, 7607 (1940) 2) K.K. Andreev, ZhPriklKhim **21**, 462–72 (1948) & CA **49**, 617 (1955)

Flashing Test. This test, designed to ascertain whether the ingredients of Black Powder are thoroughly incorporated, is conducted as follows:

Fill a small crucible (or a thimble) with the sample and by inverting transfer onto a piece of asbestos board

Touch the sample with a red hot soldering iron and observe how the powder ignites and burns

If the powder has been thoroughly blended it will "flash", or "puff off", giving only a very few sparks and leaving only some smoke marks on the board

A badly blended powder will produce many sparks and will leave specks of undecomposed K nitrate and sulfur forming a dirty residue

Powder that has been damaged by moisture will flash badly even if the ingredients had been carefully incorporated originally. This is because the saltpeter had passed partially into solution and segregated into crystals of comparatively large size
Ref: Marshall **2** (1917), 415–16

Flashless. Said of a proplnt or propelling charge which does not produce a muzzle flash in the weapon for which intended
Ref: Glossary of Ordn (1959), 119-R

Flashless Ballistite. Ballistite, a double-base proplnt (See Vol **2**, pp B8–9) can be made flashless by either incorporating Centralite and DNX oil with NG and NC or by mixing Centralite and NGu with NG and NC. The NG does not dissolve in the colloid but is distributed thru it in a state of fine subdivision. Ten or fifteen parts of NGu incorporated with 90 or 85 parts of pyrocellulose colloided with ether-alcohol gives a mixt which may be extruded thru dies and yields a flashless powder
Ref: Davis (1943), 299

Flashless Charges. See under Flash-Reducing Agents

Flashless and Cool Explosive. See Nitroguanidine (NGu) under Guanidine and Derivatives

Flashless Cordites are described in Vol **3** of Encycl, pp C532-R to C533-R, but the description of *Cordite N* was not given. For this, see Refs 1 & 2
Refs: 1) SACMS (Scientific Advisory Council of Ministry of Supplies), "Interior Ballistics", Philosophical Library, NY (1951), p 6
2) Vol **1** of Encycl, under "Albanite", p A119-R

Flashless Gunpowder. NC or NC-NG base proplnt containing up to 25% 5-Aminotetrazole had substantially reduced flash and smoke, while ballistic potential and stability were not reduced
Ref: G.C. Hale & L.F. Audrieth, USP 2480852 (1949) & CA **44**, 840 (1950)

Flashless Mortar-Type Sheet of Propellant. Flashless mortar-type smokeless proplnt with calorific value 900–1100cal/g is made from NC (12.2–13.4% N) 50–8, NG 12–44, cooling plasticizer 2–10, $(O_2NOC_2H_4)_2N(NO_2)$ 0–35%, small amt of stabilizer, small amt desensitizer and small amt inorganic salt to aid ignition. Thickness depends on burning time desired. Example of one compn used: NC (13.25% N) 55, NG 33.65, $(C_6H_5)_2NH$ 0.6, o-$C_6H_4(CO_2Et)_2$ 9.5, KNO_3 1.25 & Me cellulose 0.2 parts
Ref: A.M. Ball, USP 2577298 (1951) & CA **46**, 3764 (1952)

Flashless Nonhygroscopic (FNH) Propellant. See Vol **2** of Encycl, p C32-R, middle of column, under CANNON PROPELLANT

Flashless Propellant "Albanite" (US Navy). See Vol **1** of Encycl, p A119-R

Flashless Propellant of Gallwitz, called "G" Pulver (Ger) is mentioned in Vol **3** of Encycl, p C511-R under "Cool (or Cooled) Propellants". A complete description, including history, is given in Vol **5**, p D1536-R to D1537-R and in PATR **2510**(1958), pp Ger 70-R & Ger 71-L

Flashless Propellant "Gudolpulver" (Ger). See Vol **5** of Encycl, pp D1537-R to D1538-R and PATR **2510**(1958), pp Ger 81-L & R

Flashless Propellant of Kincaid & McGill. Triple-base + non-vol plasticizers nitroxyalkyl nitramines (See under Amines), polynitrate esters of poly(hydroxymethyl) cycloalkanones & poly(hydroxymethyl) cycloalkanols
Ref: J. Kincaid & R. McGill, USP 2698228 (1954) & CA **49**, 5846(1955)

Flashless Propellant for Naval Cannons. See "Albanite" in Vol **1**, p A119-R

Flashless Smokeless Propellant of Barsky consisted of NC 75–93, DNT (or TNT) 5–15, plasticizer & flash suppressor 2–10%. Latter was an ester of fatty acid C_6 to C_{14}, such as TEGc caprylate, TEGc caprylate-caprate or Pi-Gc caprylate
Ref: G. Barsky, USP 2439281 (1948) & CA **42**, 4349(1948)

Flashlight for Use in Photography. It is a brilliant white light, produced either by magnesium powder or by special bulbs. See Flash Photography in Vol **2** of Encycl, p C14-L & R, under CAMERAS

Flash Photography. See Vol **2**, p C14-L & R, under CAMERAS, High-Speed Photographic

Flash Photolysis. A method of conducting photochemical reactions by high intensity illumination. The illumination is from a high intensity flash of 1000–10000 J obtained by discharging a bank of condensers thru a gas (A or Kr) discharge tube (quartz) (35μF at 8000V gives ca 1000 J for 10^{-4}sec). The gas discharge tube and a reaction tube are arranged in parallel and a lamp for illumination is located at one end of the reaction tube. A suitable spectroscope is located at the other end of the reaction tube. May be obtd showing changes in short lived intermediates with time. The references cited describe the chemistry of expln of H_2 or C_2H_2 with O_2
Refs: 1) G. Porter, ProcRoySoc **A200**, 284–300(1950) & CA **45**, 10065(1951) 2) R.G.W. Norrish, DiscussionsFaradaySoc No **14**(1953) 6–22 & CA **48**, 371(1954) 3) R.G.W. Norrish et al, ProcRoySoc **A216**, 165–83(1953)

FLASH POINT (fl p) and FIRE POINT (fi p), *Flash Point* is defined in Bofors Laboratory Manual (Ref 1) as "the lowest temperature at which, under given conditions (type of apparatus, heating arrangement, etc) a liquid gives off flammable vapors which, when mixed with air under normal atmospheric pressure, flash on approaching a flame to them
Fire Point is defined and described in Item A, listed below

A. FLASH AND FIRE POINTS BY CLEVELAND OPEN CUP METHOD (COC). The Cleveland Open Cup Apparatus is described as Method **4294** (Sept 1965) of Federal Test Method Standard No 141a

The apparatus consists of a brass cup (See Fig F10), supported by a metal heating plate, ¼ inch thick and 6 inches in diameter (not shown here). In the center of the plate there is a plane depression 1/32 inch in depth, and of just sufficient diameter to fit the cup. There is also a circular opening 2-3/16 inches in diam, cut thru the plate, centering with the center of the above-mentioned depression. The plate is covered with a sheet of hard asbestos board ¼ inch thick, and of the same shape as the metal plate and with a hole cut in the center just to fit the cup. Heat may be supplied from any convenient source. The use of gas burner, electric heater, or alcohol lamp is permitted, but under no circumstances are

products of combustion or free flame allowed to come around the cup. The source of heat that does not produce local superheating shall be centered under the opening in the plate. If a flame heater is used, it shall be protected from drafts & excessive radiation by a shield that does not project above the level of the upper surface of the asbestos board

Thermometer shall be an ASTM Open Flash-Type, graduated in either centigrade (–6° to +400°C) or Fahrenheit (+20° to +760°F) degrees and conforming to the requirements for thermometer 11C or 11F, respectively, as prescribed in ASTM E1

Procedure

1. The thermometer shall be suspended or held in a vertical position by any suitable device. The bottom of the bulb shall be ¼ inch (0.635cm) from the bottom of the cup, and above a point half way between the center and back of the cup

2. The cup shall be filled with the sample to be tested in such a manner that the top of the meniscus is exactly at the filling line at room temperature. The surface of the sample shall be free from bubbles. There shall be none of the sample above the filling line or on the outside of the apparatus

3. The test flame shall be approx 5/32 inch (0.397cm) in diameter

4. The test flame shall be applied as the temperature read on the thermometer reaches each successive 5°F (2.8°C) mark. The flame shall pass in a straight line (or on the circumference of a circle having a radius of at least 6 inches) across the center of the cup and at right angles to the diam passing thru the thermometer. The test flame shall, while passing across the surface of the sample, be in the plane of the upper edge of the cup. The time for the passage of the test flame across the cup shall be approx 1 second

5. The sample shall be heated at a rate not exceeding 30°F (16.7°C) per minute temp rise, until a point is reached approx 100°F (55.6°C) below the probable fl p of the sample. Thereafter the rate of heating shall be decreased, and for at least the last 50°F (27.8°C) before the fl p is reached, the rate shall be not less than 9°F (5°C) nor more than 11°F (6.1°C) per minute

6. Flash Point. The flash point shall be taken as the temp read on the thermometer

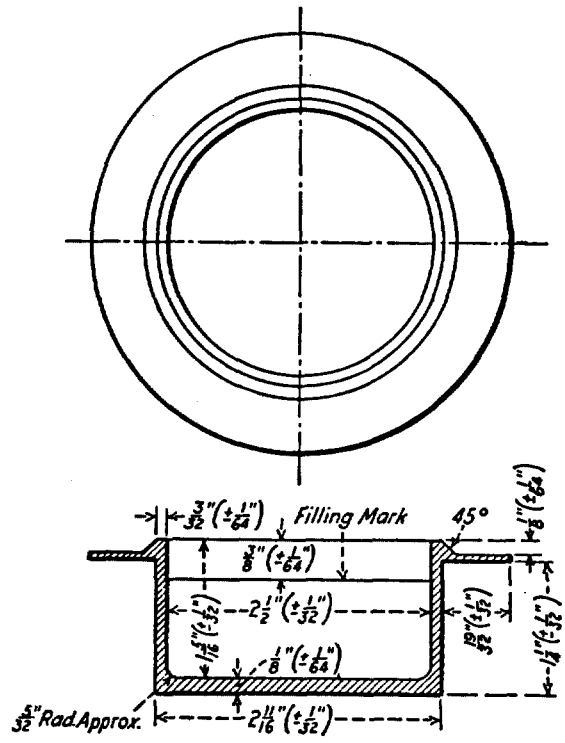


FIG F10 CLEVELAND OPEN CUP

when a flash appears at any point on the surface of the sample. The true flash must not be confused with a bluish halo that sometimes surrounds the test flame

7. Fire Point. After determining the fl p, the heating shall be continued at the specified rate of 9° to 11°F (5° to 6.1°C) per minute, and application of the test flame shall be made at the specified intervals until the sample ignites and continues to burn for a period of at least 5 seconds. The method of application of the flame shall be the same as for fl p. The temp read at the time of the flame application which causes burning for a period of 5 seconds or more shall be recorded as the *fire point*

Notes:

a. The following facilities are of great importance and should be carefully observed. Laboratory conditions shall be such that flash and fire point tests are made in a room

or compartment free from air currents. Care shall be observed to avoid disturbing the vapors evolved in the cup while heating, either by careless breathing or unnecessary movements near the flash cup. It is desirable that the room or compartment be darkened sufficiently so that the flash can be readily detected

b. Results shall not differ from each other by more than the following:

Flash or fire point (°F)	Repeatability, one operator and apparatus (°F)	Reproducibility, different operators and apparatus (°F)
175 to 550	5	10
Over 550	10	15

FLASH POINT OF LIQUIDS BY TAG OPEN CUP APPARATUS (ASTM Designation D1310-72)

1. Scope

1.1 This method covers the determination of flash points by Tag open-cup apparatus of liquids having flash points between 0 and 325 F (–17.8 and 168 C)

1.2 This method, when applied to paints and resin solutions which tend to skin over or which are very viscous, gives less reproducible results than when applied to solvents

Note 1: A value of 80 F (26.7 C) determined by this method has been established by the US Department of Transportation as the maximum value for classifying liquids as flammable. Materials having a flash point at or below 80 F (26.7 C) must be identified with an ICC Red Label for interstate shipment by common carrier within the United States. In addition, certain regulations on types of shipping containers apply to materials flashing at 20 F (–6.7 C) or below

For details of ICC shipping regulations, see Tariff No 19, issued August 5, 1966, by T.C. George, Agent, 63 Vesey St, New York, NY 10007, publishing Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles by Land and Water in Rail Freight Service and by Motor Vehicle (highway and water) including Specifications for Shipping Containers. (Regulations for transportation in rail express and rail baggage services are also included for information)

Note: Shipping regulations are now updated annually by the Department of Transportation. Be sure to use the latest regulation. For more information on shipping regulations, see EXPLOSIVES, NONMILITARY (COMMERCIAL) (Papers and Reports Listed in Chronological Order), Item 87, "On Shipping Explosives", this Vol of Encycl, p E468

2. Apparatus (Appendix A1)

A1.1 Tag Open-Cup Tester, shown in Fig F11. It consists of the following parts, which must conform to the dimensions shown, and have the additional characteristics as noted:

Metric Equivalents	
in.	mm
2-13/16	71.4
3-3/16	80.9
6-7/8	174.6

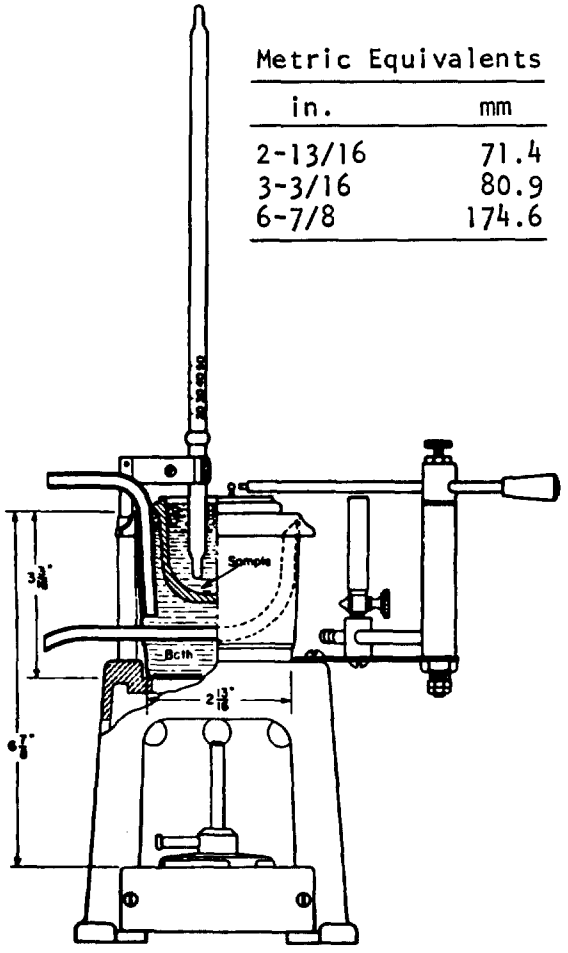


FIG F11 TAG OPEN CUP TESTER

A1.1.1 Copper Bath, preferably equipped with a constant-level overflow so placed as to maintain the bath liquid level 1/8 inch (3.2mm) below the rim of the glass cup

A1.1.2 Thermometer Holder, supplied with the tester as shown in Fig. It shall support the thermometer firmly in a vertical position. Thermometers must conform to ASTM Specification

A1.1.3 Glass Test Cup is of molded clear glass, annealed, heat-resistant, and free from surface defects

A1.1.1.4 Leveling Device, or gage, for proper adjustment of the liquid level in the cup. This shall be made of polished aluminum or stainless steel 1/8 inch (3.2mm) (0.125±0.003 inch (3.2±0.07mm) thick, with two projections for adjusting the liquid level in the glass cup to exactly 1/8 inch below the top edge or rim of the cup. This leveling device may also be used to adjust the size of the test flame and for gaging the height of the taper above the edge of the cup

A1.1.5 "Micro" or Small Gas Burner, of suitable dimensions for heating the bath. A screw clamp may be used to help regulate the gas. A small electric heater controlled by a variable power transformer may be used

A1.1.6 Ignition Taper, which is a small, straight, blow-pipe type gas burner. The tip of the taper should be approx 1/16 inch (1.6mm) in diam and the orifice should be 1/32 inch 0.8mm) in diam. The ignition taper should be maintained in a fixed horizontal plane above the test cup by means of a swivel device so that the test flame passes on the circumference of a circle having a radius of at least 6 inches (152.4mm)

A1.1.7 Draft Shield, consisting of two rectangular sheets of noncombustible material, 24 by 28 inches (610 by 710mm), are fastened together along the 28-inch (710mm) side, preferably by hinges. A triangular sheet, 24 by 24 by 34 inches (610 by 610 by 860mm), is fastened by hinges to one of the lateral sheets (to form a top when shield is open). The interior of the draft shield shall be painted a flat black. A draft-free fume hood may be used

Two 500-ml flasks with rubber stoppers

6. *Procedure*

6.1 For flash points below 55 F (13 C) or above 140 F (60 C), use as bath liquid a 1 + 1 mixture of water and ethylene glycol. For flash points between 55 F (13 C) and 140 F (60 C), either water or water-glycol mixture may be used as bath liquid (Note 4). The temp of the liquid in the bath shall be at least 20 F (11 C) below the expected fl p at the time of introduction of the sample into the test cup. Do not cool the bath liquid by direct contact with carbon dioxide or "dry ice". Place the test cup in position in the bath

Note 4: Due to possible difficulty in maintaining the prescribed rate of temp rise and due to the formation of ice on the lid, results by this method for samples having fl p's below 32 F (0 C) may be somewhat unreliable. Trouble due to ice formation on the slide may be minimized by carefully lubricating the slide shutter with high-vacuum silicone lubricant

6.2 Using a graduate and taking care to avoid wetting the cup above the final liquid level, measure 50±0.5ml of the sample into the cup, both the sample and graduate being precooled, if necessary, so that the sample temp at the time of measurement will be 80±10 F (27±5.6 C) or at least 20 F (11 C) below the expected flash point, whichever is lower. It is essential that the sample temp be maintained at least 20 F (11 C) below the expected fl p during the transfers from the sample container to the graduate and from the graduate to the test cup. Destroy air bubbles on the surface of the sample. Wipe the inside of the cover with a clean cloth or absorbent tissue paper; then attach the lid, with the thermometer in place, to the bath collar

6.3 Light the test flame, adjusting it to the size of the small bead on the cover. Operate the mechanism on the cover in such a manner as to introduce the test flame into the vapor space of the cup, and immediately bring it up again. The time consumed for the full operation shall be about 1 sec, or the time required to pronounce distinctly the words "thousand and one". Avoid any jerkiness in the operation of depressing and raising the test flame

6.4 Flash Points Below 140 F (60 C). If the fl p of the sample is known to be below 140 F (60 C), apply and adjust the heat so that the temp of the sample will rise at a rate of 2 F (C)/min±6 secs. When the temp of the sample in the test cup is 10 F (5.6 C) below its expected fl p, apply the test flame in the manner just described in 6.3, and repeat the application of the test flame after each 1 F (0.6 C) rise in temp of the sample

6.5 Flash Points at or above 140 F (60 C). If the fl p of the sample is known to be 140 F or higher, apply and adjust the heat so that the temperature of the sample will rise at a rate of 5 F (3 C)/min±6 secs. When the temp of the sample in the test cup is 10 F (5.6 C) below its expected fl p, apply the test flame in the manner described in 6.3, and repeat the application of the test flame after each 2 F (1 C) rise in temperature of the sample, at each temperature reading that is a multiple of 2 F (1 C)

6.6 When the test flame application causes a distinct flash in the interior of the cup, observe and record the temperature of the sample as the fl p. Do not confuse the true flash with the bluish halo which sometimes surrounds the test flame at applications immediately preceding the actual flash

6.7 Discontinue the test and remove the source of heat. Lift the lid and wipe off the thermometer bulb. Remove the sample cup, empty, and wipe dry

6.8 If, at any time between the first introduction of the test flame and the observation of the flash point, the rise in temp of the sample is not within the specified rate, or if the actual flash point differs from the expected flash point by an amt greater than 4 F (2 C), discard the result and repeat the test, adjusting the source of heat to secure the proper rate of temp rise and/or using a modified "expected flash point", as required

Note 5: Never make a repeat test on the same portion of sample once used- always take a fresh portion of sample for each test

7. *Correction for Barometric Pressure*

7.1 Observe and record the barometric pressure at the time of the tests. When the pressure differs from 760mm Hg, correct the fl p by means of the following equation, the corrected fl p being recorded to the nearest whole number

Corrected flash point = F + 0.06 (760 − P)

3. *Summary of Method*

3.1 The sample is placed in the cup of a Tag Open Tester, and heated at a slow but constant rate. A small test flame is passed at a uniform rate across the cup at specified intervals. The flash point is taken as the lowest temp at which application of the test flame causes the vapor at the surface of the liquid to flash. In the case of less volatile materials, the vapors may ignite but not continue to burn. With more volatile materials, the fl p and fi p may occur simultaneously

4. *Apparatus* – described under 2

5. *Materials*

5.1 Water–Glycol Solution (1 + 1), for flash points from 0 to 200 F (−17.8 to 93.3 C)

5.2 Solid Carbon Dioxide-Acetone or other coolant

5.3 Silicone Fluid, inert, high-boiling, for fl p's from 200 to 325 F (93.3 to 168 C)

5.4 n-Heptane, for determination of fl p's from 0 to 60 F (−17.8 to 15.6 C). See Appendix A2 for specifications

5.5 p-Xylene, for determination of fl p's from 60 to 200 F (15.6 to 93.3 C). See Appendix A2 for specifications

5.6 Isopropyl Alcohol, for determination of fl p's from 60 to 200 F (15.6 to 93.3 C). See Appendix A2 for specifications

5.7 Diethylene Glycol, for determination of fl p's from 200 to 325 F (93.3 to 168 C)

Appendix A2

A2.1 Specifications for n-Heptane; Flash Point Check Grade

A2.1.1 n-Heptane shall conform to the following requirements:

Density at 20 C – 0.6830±0.00015 when determined in accordance with Method D1217

Refractive Index $n_D^{20\,C}$ – 1.38770±0.00015 when determined in accordance with Method D1218

Freezing Point – −90.71 C min, when determined in accordance with Method D1015

Distillation (760mm) – 50% recovered at 98.427±0.025 C. Differential, 80% recovered minus 20% recovered – 0.20 C, max

Note: For equipment and method used, see C.S. Dussinger et al in JResearchJRMSB NatBurStandards 44(3), 309–10 (1950)

A2.2 Specifications for p-Xylene; Flash Point Check Grade

A2.2.1 p-Xylene shall conform to the following requirements:

Specific gravity 15.56/15.56 C – 0.860 min, 0.866 max

Boiling range – 2 C max from start to dry point, when tested in accordance with Method D 850, or the Method D 1078. The range shall include the boiling point of pure p-xylene, which is 138.35 C (281.03 F)

Purity – 95% min (Freezing point of 11.23 C min) calculated in accordance with Method D 1016, from the experimentally determined freezing point, measured by Method D 1015

A2.3 Specifications for Isopropanol 91%; Flash Point Check Grade

A2.3.1 Isopropanol shall conform to the following requirements:

Specific gravity – 0.8175 to 0.8185 at 20/20 C as determined by means of a calibrated pycnometer

Distillation range – shall entirely distill within a 1.0 C range which shall include the temp 80.4 as determined by Method D 1078

A2.4 Specifications for Diethylene Glycol; Flash Point Check Grade

A2.4.1 Diethylene glycol shall conform to the following requirements:

Specific gravity – 1.1170 to 1.1200 at 20/20 C as determined by means of a calibrated pycnometer

Distillation range – shall entirely distill within a 5.0 range which shall include the temp 245.8 C as determined by Method D 1078

Water – not more than 0.2% as determined by Method D 1364

6. *Assembly and Preparation of Apparatus*

6.1 Place the tester in a level position on a solid table free of vibration, in a location free of perceptible draft, and in a dim light. Maintain a room temp of 75±5 F (24±3 C) thruout the test

6.2 Adjust the horizontal and vertical positions of the taper so that the jet passes on the circumference of a circle having a radius of at least 6 inches (152.4mm). The jet should pass across the center of the cup at right angles to a diam passing thru the thermometer, and in a plane 1/8 inch (3.2mm) above the upper edge of the cup as measured from the center of the orifice

6.3 Using the leveling device as a gage, adjust the height of the taper so that the center of the orifice is exactly 1/8 inch (3.2mm) above the top edge of the glass cup when it is in place. It is imperative that this adjustment be made as accurately as possible. Raising or lowering the taper can be achieved by bending it slightly or preferably by adding and removing thin metal shims as required from between the taper and the vertical supporting member of the swivel holder (See Note 7)

6.4 With the glass cup in place in the bath, adjust the thermometer holder so that the thermometer is supported firmly in a vertical position half way between the center and edge of the cup and the pivot of the taper. Place the thermometer so that the bottom of the bulb is 1/4 inch (6.4mm) from the inner bottom of the cup

6.5 Set the draft shield around the tester so that the sides form right angles with each other and the tester is well toward the back of the shield

7. *Procedure*

7.1 Flash Points from 0 to 60 F (–17.8 to 15.6 C):

Note 2: Caution – Meticulous attention to all details relating to the taper, size of taper flame, rate of temperature increase, and rate of passing the taper over the sample is necessary for good results

7.1.1 Equip two 500-ml flasks with rubber stoppers thru which are inserted ASTM 33F thermometers. Cool a quantity of 1 to 1 water-glycol solution in one stoppered 500-ml flask to approx –20 F (–28.9 C) by immersing the flask in a solid carbon dioxide-acetone bath or other coolant. Use extreme care not to contaminate the water-glycol solution with either acetone or carbon dioxide

7.1.2 Pour the cooled water-glycol solution into the tester bath to a predetermined level 1/8 inch (3.2mm) below the top when the cup is in place. An overflow pipe is desirable for controlling the liquid level in the bath

7.1.3 At the same time the water-glycol

coolant is being chilled, cool a portion of the sample to approx −10 F (−23.3 C) in the second stoppered 500-ml flask. If solid carbon dioxide and acetone or other volatile solvents are used as a coolant, extreme care must be exercised to avoid contamination of the sample. Cool the glass cup and place it in the bath. Position the appropriate thermometer as described in 5.4 and fill the cup with cooled sample to a depth approx 1/8 inch (3.2mm) below the edge as determined by the leveling device

7.1.4 Light the ignition flame and adjust it to form a flame of spherical shape matching in size the 5/32-inch (4.0mm) sphere on the apparatus or the 5/32-inch hole in the leveling device

Note 3: Remove all bubbles from the surface of the sample liquid before starting a determination

7.1.5 Final adjustment of the sample level in the cup is made when the temperature is 20 F (11 C) below the anticipated flash point. Two trial determinations may be necessary to select the proper temperature at which to adjust the liquid level. A hypodermic syringe or medicine dropper provides a convenient means of adding or removing sample from the cup

7.1.6 Allow the temperature of the sample to increase spontaneously without applying any heat until the rate of temperature rise decreases to 2 F (1 C)/min. At this point apply heat to maintain an increase in temperature at a rate of 2±0.5 F (±0.25 C)/min

Note 4: With viscous materials, this rate of heating cannot always be maintained

7.1.7 Determine the approx flash point by passing the taper flame across the sample at intervals of 2 F (1 C). The first pass of the taper flame should be made immediately after the final adjustment of the sample level, as in 6.1.5. The time required to pass the ignition flame across the surface of the sample should be 1 sec. Each pass must be in one direction only and the taper should be kept in the "off" position at one or the other end of the swing, except when the flame is applied to the sample. In case the sample tends to "creep" over the edge of the cup, carefully wipe the edge with absorbant tissue to remove frost and liquid just prior to passage of the taper over the cup

Note 5: When determining the flash point of viscous liquids and those liquids which tend to form a surface film, the following procedure is suggested: Insert to a depth of about ½ inch (12.7mm) in approx a vertical position, the end of a stirring rod beginning about 15 sec before the taper is passed over the surface. Move the rod from side to side of the cup for three or four complete passes following approx the path of the taper, remove, and make the test

7.1.8 Continue with 7.4

7.2 *Flash Points from 60 to 200 F (15.6 to 93.3 C) (See Notes 2, 3, 4 and 5):*

7.2.1 Run cold water, brine or water-glycol solution into the bath to a predetermined level which will fill the bath to 1/8 inch (3.2mm) below the top when the cup is in place. The bath liquid should be at least 30 F (17 C) below the anticipated flash point

7.2.2 If necessary, cool a portion of the sample to at least 20 F (11 C) below the anticipated flash point. Exercise adequate care to avoid contamination of the sample with coolant liquid or vapors. Fill the glass cup with the cooled sample to a depth approx 1/8 inch (3.2mm) below the edge with the proper thermometer (See Table 1) positioned as described in 6.4

7.2.3 For final adjustment of the sample level, see 7.1.5

7.2.4 Light the ignition flame and adjust it as described in 7.1.4

7.2.5 Apply heat to the liquid bath and adjust so that the temperature of the sample increases at a rate of 2±0.5 F (1±0.25 C)/min

7.2.6 Determine the approx flash point by passing the taper flame across the sample at intervals of 2 F (1 C) as described in 7.1.7

7.2.7 Continue with 7.4

7.3 *Procedure for Flash Points from 200 to 325 F (93.3 to 168 C) (See Notes 2, 3, 4 and 5):*

7.3.1 Place a high-boiling inert silicone fluid in the bath to a predetermined level which will fill the bath to 1/8 inch (3.2mm) below the top when the cup is in place

7.3.2 With the appropriate thermometer (See Table 1) properly positioned (6.4), fill the glass cup with sample at room temperature to a depth slightly more than 1/8 inch (3.2mm)

below the edge as determined by the leveling device

TABLE 1 Thermometers

For Tests	Below 40 F (4 C)	At 40 to 120 F (4 to 49 C)	Above 120 F (49 C)
Use ASTM Thermometer*	57 F or 57 C	9 F or 9 C 57 F or 57 C	9 F or 9 C

* Complete specifications for these thermometers are given in ASTM Specification E 1, for ASTM Thermometers

7.3.3 For final adjustment of the sample level, see 7.1.5

7.3.4 *Light the ignition flame and adjust it* as described in 7.1.4

7.3.5 Apply full heat to the liquid bath and when the temperature of the sample reaches 190 F (87.8 C), adjust the heat input so that the temp of the sample increases at a rate of 2±0.5 F (1±0.25 C)/min

Note 6: The heaters on some testers do not have sufficient capacity to maintain the proper rate of heating when the temperature approaches 250 F (121.1 C) or above. The heat input to the liquid bath may be increased if necessary by using a variable transformer to increase the voltage on the heater or by wrapping the bath with electrical heating tape. The application of suitable insulation to the outdide of the bath to prevent heat loss is also permissible. The important factor is to maintain the rate of temp increase of the sample at 2±0.5F (1±0.25 C)/min

7.3.6 Determine the approximate flash point by passing the taper flame across the sample at intervals of 2 F (1 C) as described in 7.1.7

7.4 *Recorded Test:*

7.4.1 After the initial test to determine the approx flash point of the material, repeat the procedure by cooling a fresh portion of the sample, the glass cup, the bath solution, and the thermometer to more than 20 F (11 C) below the approx flash point. When the temp of the sample is exactly 20 F below the approx flash point, the center of the liquid level should be adjusted to 1/8 inch (3.2mm) below the upper edge of the cup as determined with the leveling device placed across the diam of the cup

7.4.2 Resume heating, or allow the temp to rise spontaneously in the case of material flashing below 60 F (15.6 C). Following the instructions given in 6.1, pass the taper flame across the sample at two intervals of 5 F (3 C) and then at intervals of 2 F (1 C) until the flash point occurs

7.4.3 Determine and record not less than three test values

8. *Standardization and Calculation*

8.1 *Flash Points from 0 to 60 F (−17.8 to 15.6 C):*

8.1.1 Make at least five determinations of the flash point of standard n-heptane which meets the specifications set forth in Appendix A2. Average these values. If the average differs from 23 F (−4 C) by more than ±3 F (±1.7 C), adjust the height of the taper and repeat the standardization (Note 7)

8.1.2 Calculate the correction factor as follows:

$$\text{Correction factor} = 23 - A$$

where: A = average observed flash point of n-heptane

8.1.3 Apply this correction to all flash point determinations between 0 and 60 F (−17.8 to 15.6 C). Round off corrections to the nearest whole number according to Recommended Practice E 29

8.2 *Flash Points from 60 to 200 F (15.6 to 93.3 C):*

8.2.1 Make determinations in triplicate on the flash point of standard *p*-xylene and of standard isopropyl alcohol which meet specifications set forth in Appendix A2. Average these values for each compound. If the difference between the values for these two compounds is less than 15 F (8.5 C) or more than 27 F (16 C), repeat the determinations or obtain fresh standards (Note 7)

8.2.2 Calculate a correction factor as follows:

$$X = 92 - A$$
$$Y = 71 - B$$
$$\text{Correction} = (X + Y) / 2$$

where: A = observed flash of *p*-xylene, and
B = observed flash of isopropyl alcohol

8.2.3 Apply this correction to all flash point detns between 60 and 200 F (15.6 and 93.3 C). Round off corrections to the nearest whole number according to Recommended Practice E 29

8.3 *Flash Points from 200 to 325 F (93.3 to 168 C):*

8.3.1 Make at least five detns of the flash point of standard diethylene glycol which meets the specifications set forth in *Appendix A2*. Average these values and if the average differs from 295 F (146 C) by more than ±10 F (±5.5 C), adjust the height of the taper and repeat the standardization

Note 7: The height of the taper arm is very important. Raising the taper 0.01 inch (0.25 mm) increases the flash about 2 F (11 C). Therefore, it is is suspected that the taper arm has been jarred or bent, the apparatus should be recalibrated. Each unit of apparatus should have its calibration checked about once a week if in constant use, or on each occasion of use if used only occasionally

8.3.2 Calculate the correction factor as follows:

$$\text{Correction} = 295 - A$$

where: A = average observed flash point of diethylene glycol

8.3.3 Apply this correction to all flash point determinations between 200 and 325 F (93.3 and 168 C). Round off corrections to the nearest whole number according to Recommended Practice E 29

Note 8: The calibration procedure provided in this method will cancel out the effect of barometric pressure if calibration and tests are run at same pressure

9. Report

9.1 The average of not less than three corrected recorded tests, other than the initial test, shall be reported to the nearest 1 F (0.5 C). Three multiple runs are acceptable for averaging if the difference between the extreme values does not exceed 7 F (4 C) (95% confidence level)

10. Precision[7]

10.1 The following criteria should be used for judging the acceptability of results between 0 and 200 F (−17.8 and 93.3 C) at a 95% confidence level:

10.1.1 Repeatability – Two results (each the average of at least three detns) obtained by the same analyst will differ, on the average, by approx 1.5 F (0.8 C). They should be considered suspect if the difference exceeds 4 F (2.2 C).

10.1.2 Reproducibility – Two results (each the average of at least three detns) reported by analysts in different laboratories will differ, on the average, by approx 2.5 F (1.4 C). They should be considered suspect if the difference exceeds 7 F (4 C)

10.2 The following criteria should be used for judging the acceptability of results between 200 and 325 F (93.3 and 168 C) at a 95% confidence level:

10.2.1 Repeatability – Two results (each the average of at least three detns) obtained by the same analyst will differ, on the average, by approx 3.0 F (1.7 C). They should be considered suspect if the difference exceeds 9 F (5 C)

10.2.2 Reproducibility – Two results (each the average of at least three detns) reported by analysts in different laboratories will differ, on the average, by approx 4.0 F (2.2 C). They should be considered suspect if the difference exceeds 12 F (7 C)

10.3 On the basis of an interlaboratory test of the method using viscous, heavily-pigmented materials which tended to form a surface film, the within-laboratory standard deviation was found to be 3 F (1.7 C) and the between-laboratory standard deviation was found to be 7 F (3.9 C). Based on these standard deviations, the following criteria should be used for judging the acceptability of results at a 95% confidence level:

10.3.1 Repeatability – Two results, each the mean of three detns, obtained by a single operator on different days should be considered suspect if they differ by more than 9 F (5 C)

10.3.2 Reproducibility – Two results, each the mean of three detns, obtained by operators in different laboratories should be considered suspect if they differ by more than 24 F (13.3 C)

FLASH POINT OF LIQUIDS BY TAG CLOSED TESTER (ASTM Designation D56–70)

1. Scope

1.1 This method covers the detn of the flash point, by Tag closed tester, of liquids with a viscosity of below 45 SUS at 100 F (37.8 C) and a fl pt below 200 F (93 C) except cut-back asphalts and those liquids which tend to form a surface film under test conditions

Note 1: For the closed cup fl pt of liquids with

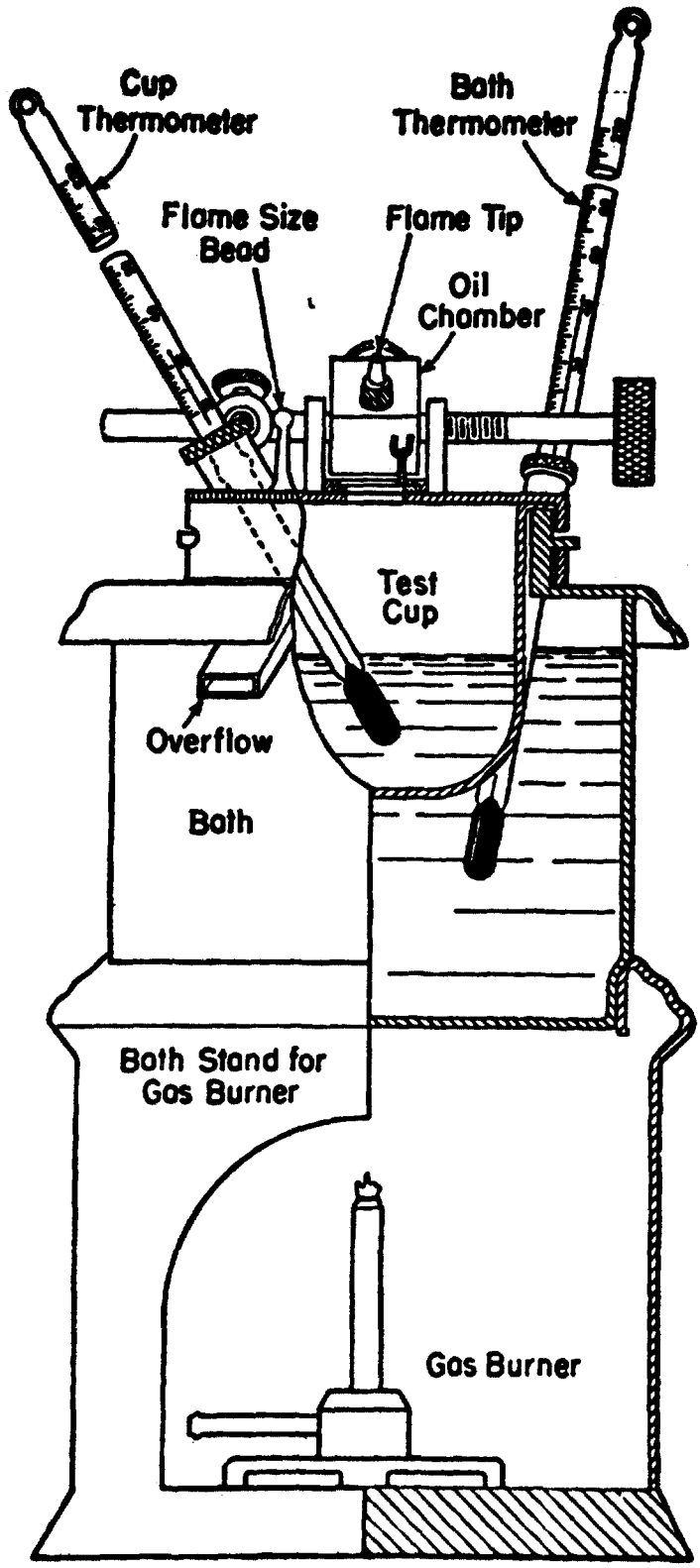


FIG F12 TAG CLOSED TESTER

a viscosity of 45 SUS or more at 100 F (37.8C) or a fl pt of 200 F or higher, use ASTM Method D 93. Test for Flash Point by Pensky-Martens Closed Tester. For cut-back asphalts refer to ASTM Method D 1310, Test for Flash Point of Liquids by Tag Open-Cup Apparatus

2. Summary of Method

2.1 The sample is placed in the cup of the tester and, with the lid closed, heated at a specified constant rate. A small flame of specified size is directed into the cup at regular intervals. The fl pt is taken as the lowest temp at which application of the test flame causes the vapor above the sample to ignite

3. Apparatus

3.1 Tag Closed Tester – The apparatus is shown in Fig 11 and described in detail in *Appendix A1:* Refer to Appendix A2 for directions for checking the condition and operation of the tester

3.2 Shield – A shield 18 inches (460mm) square and 24 inches (610mm) high, open in front, is recommended

3.3 Thermometers – For the test cup thermometer, use one as prescribed in Table 1. For the bath thermometer, any convenient type which has adequately open scale covering the required range may be used; it is often convenient to use the same type of thermometer as used in the test cup

Note 2: Whenever thermometers complying with ASTM requirements are not available, thermometers complying with the requirements for The Institute of Petroleum thermometer IP 15F PM-Low may be used

Note 3: There are automatic flash point testers available and in use which may be advantageous in the saving of testing time, permit the use of smaller samples, and other factors which may merit their use. If automatic testers are used, the user must be sure that all of the manufacturer's instructions for calibrating, adjusting and operating the instrument are followed. In any cases of dispute, the fl pt as detd manually shall be considered the referee test

4. Sample

4.1 Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Containers shall not be opened unnecessarily and transfers shall not be made unless the sample temp is at least 20 F (11 C) below the expected fl pt. Samples in leaky containers shall be discarded

5. Preparation of Apparatus

5.1 Support the tester on a level steady table. Unless tests are made in a draft-free room or compartment, surround the tester on three sides by the shield for protection from drafts. Tests made in a laboratory draft hood or near ventilators are not to be relied upon

5.2 Gas is recommended for the test flame. If gas is not available, insert a wick of cotton in the burner tip, place small quantity of cotton waste in the chamber to which the burner tip is attached, and fill the chamber with signal, sperm, or lard oil

8. Precision

8.1 The following criteria should be used for the acceptability of results (95% probability)

8.1.1 Repeatability – Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amounts:

Flash Point	Reproducibility
Below 55 F (13 F)	6 F (3.3 C)
55 F (13 C) to 139 F (59 C)	4 F (2.2 C)
140 F (60 C) to 199 F (93 C)	6 F (3.3 C)

FLASH POINT OF LIQUIDS BY PENSKY-MARTENS CLOSED TESTER

(ASTM Designation E134–68)

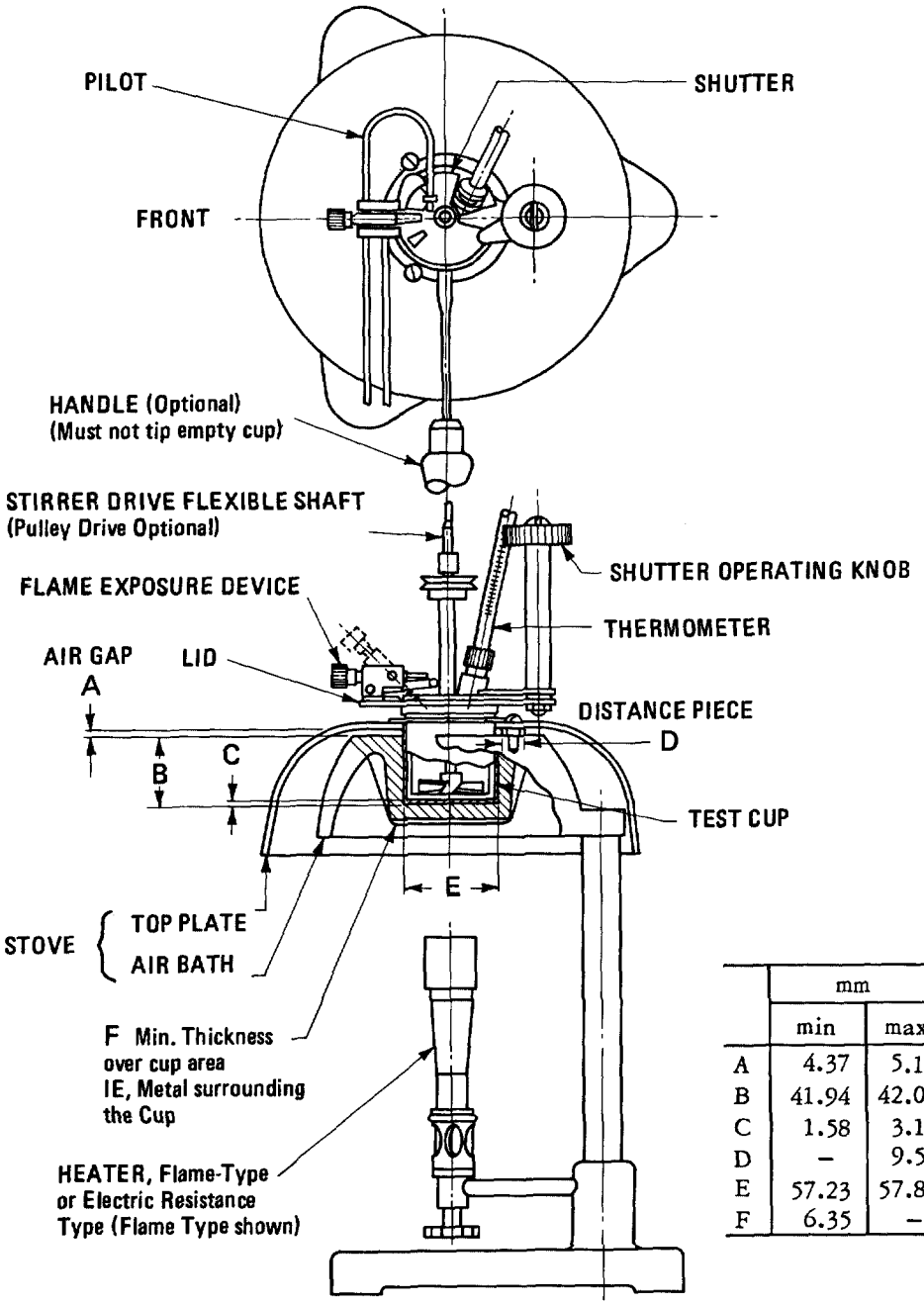
1. Scope

1.1 This specification defines the Pensky-Martens Closed Flash Tester as used in ASTM Method D93, Test for Flash Point by Pensky-Martens Closed Tester

Note 1: The values stated in US customary units are to be regarded as the standard. The metric equivalents of US customary units may be approximate

2. Apparatus Assembly

2.1 A typical assembly of the apparatus, gas heated, is shown in Fig F13. The apparatus shall consist of a test cup, lid, and stove conforming to the requirements given in Sections 3 to 5



	mm		inches	
	min	max	min	max
A	4.37	5.16	0.172	0.203
B	41.94	42.06	1.651	1.656
C	1.58	3.18	0.062	0.125
D	–	9.52	–	0.375
E	57.23	57.86	2.253	2.278
F	6.35	–	0.25	–

FIG F13 Pensky-Martens Closed Tester

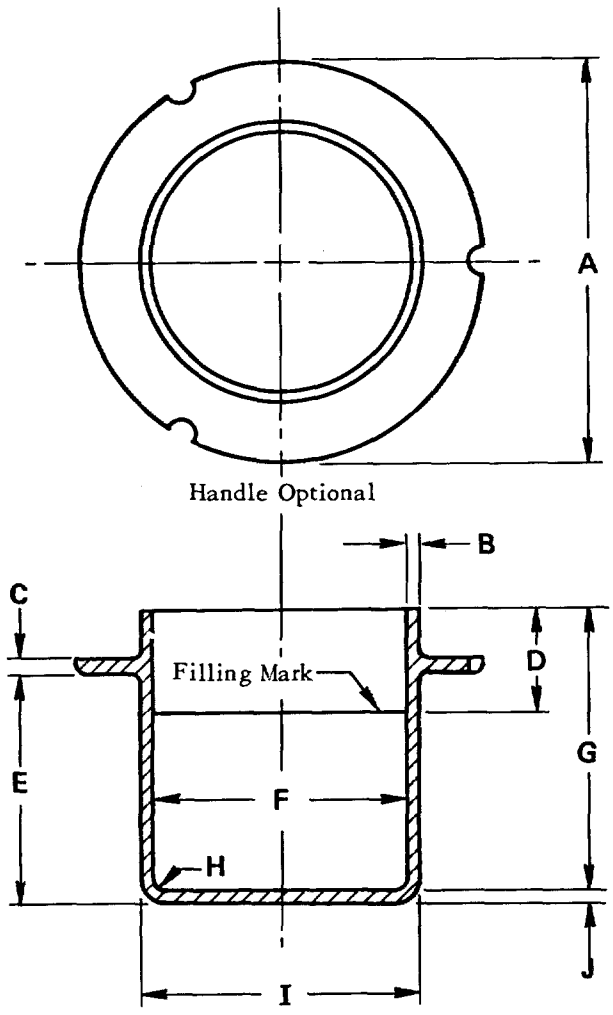


FIG F14 Test Cup

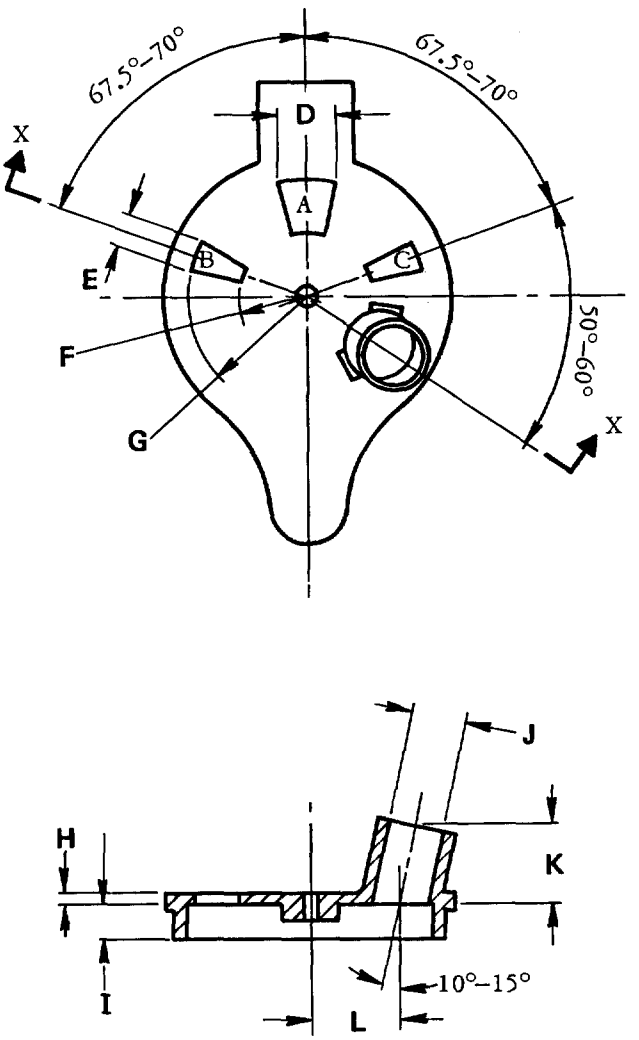
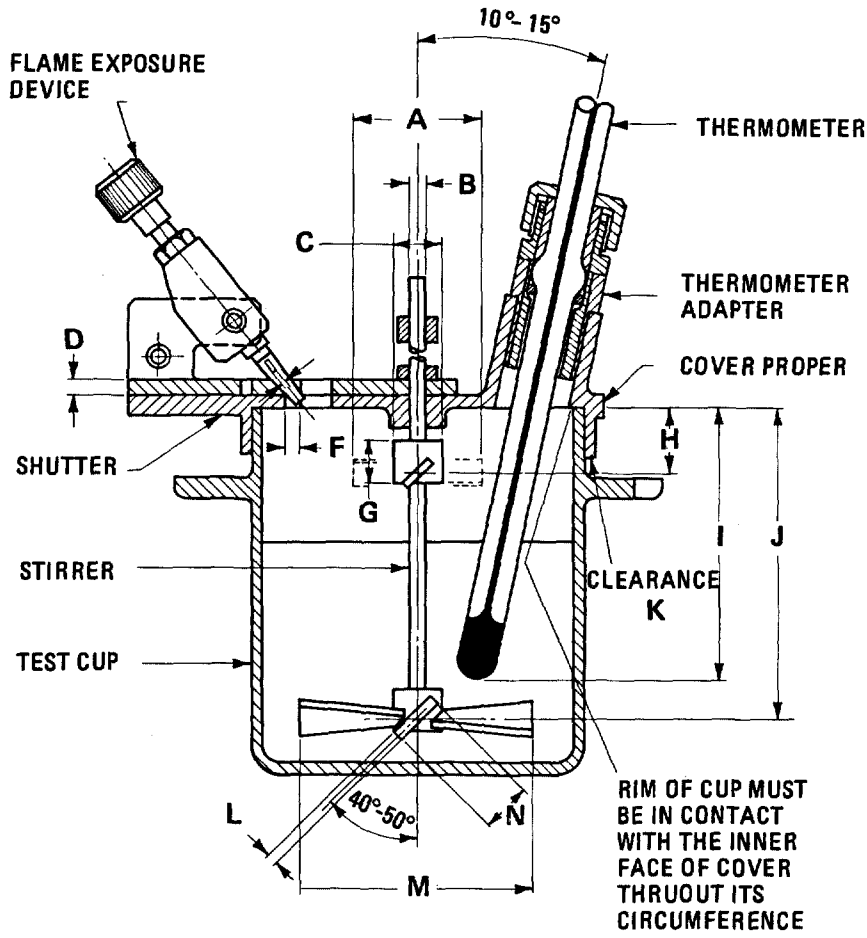


FIG F15 Cover Proper

	mm		inches	
	min	max	min	max
A	79.0	79.8	3.11	3.14
B	1.0	–	0.04	–
C	2.8	3.6	0.11	0.14
D	21.72	21.84	0.855	0.860
E	45.47	45.72	1.790	1.800
F	50.72	50.85	1.997	2.002
G	55.75	56.00	2.195	2.205
H	3.8	4.0	0.15	0.16
I	53.90	54.02	2.122	2.127
J	2.29	2.54	0.090	0.100

	mm		inches	
	min	max	min	max
D	12.7	13.5	0.50	0.53
E	4.8	5.6	0.19	0.22
F	13.5	14.3	0.53	0.56
G	23.8	24.6	0.94	0.97
H	1.2	2.0	0.05	0.08
I	7.9	–	0.31	–
J	12.27	12.32	0.483	0.485
K	16.38	16.64	0.645	0.655
L	18.65	19.45	0.734	0.766



	mm		inches	
	min	max	min	max
A	18.3	19.8	0.72	0.78
B	2.38	3.18	0.094	0.125
C	7.6	8.4	0.30	0.33
D	2.0	2.8	0.08	0.11
E	0.69	0.79	0.027	0.031
F	2.0	2.8	0.08	0.11
G	6.4	10.4	0.25	0.11
H	9.6	11.2	0.38	0.44
I	43.0	46.0	1.69	1.81
J	50.0	51.6	1.97	2.03
K	–	0.36	–	0.014
L	1.22	2.06	0.048	0.08
M	31.8	44.4	1.25	1.75
N	7.6	8.4	0.30	0.33

FIG F16

FIG F16 Test Cup and Lid Assembly

3. Cup

3.1 The cup shall be of brass, or other non-rusting metal of equivalent heat conductivity, and shall conform to the dimensional requirements in Fig F14. The flange shall be equipped with devices for locating the position of the cup in the stove. A handle attached to the flange of the cup is a desirable accessory

4. Lid

4.1 Cover Proper – The cover shown in Fig F15 shall be of brass (Section 3), and shall have a rim projecting downward almost to the flange of the cup. The rim shall fit the outside of the cup with a clearance not exceeding 0.36mm (0.014 inch). There shall be a locating device engaging with a corresponding device on the stove. The four openings in the cover A, B, C and D are shown in Fig F15

4.2 Shutter – The lid shall be equipped with a brass (Section 3) shutter (Fig F16), operating on the plane of the upper surface of the cover. The shutter shall be so shaped and mounted that it rotates on the axis of the horizontal center of the lid between two stops so placed, that when in one extreme position, the openings A, B and C of the lid are completely closed, and when in the other extreme position, these openings are completely opened

4.2.1 The mechanism operating the shutter should be the spring type and constructed so that when at rest the shutter shall exactly close the three openings. When operated to the other extreme, the three lid openings shall be exactly open and the tip of the exposure tube shall be fully depressed

4.3 Flame Exposure Device – The flame exposure device (Fig F16) shall have a tip with an opening 0.69 to 0.79mm (0.027 to 0.031 inch) in diam. This tip shall be made preferably of stainless steel, although it may be fabricated of other suitable metals. The flame exposure device shall be equipped with an operating mechanism which, when the shutter is in the "open" position, depresses the tip so that the center of the orifice is between the planes of the under and upper surfaces of the lid proper at a point on a radius passing thru the center of the larger opening A (Fig F15)

4.4 Pilot Flame – A pilot flame shall be provided for automatic relighting of the exposure flame. A bead 4mm (5/32 inch) in diam may be mounted on the lid so that the size of the test flame can be regulated by comparison

4.5 Stirring Device – The lid shall be equipped with a stirring device (Fig F13), mounted in the center of the lid and carrying two two-bladed metal propellers. A stirrer shaft may be coupled to the motor by a flexible shaft, or a suitable arrangement of pulleys

5. Stove

5.1 Heat shall be supplied to the cup by means of a properly designed stove which is equivalent to an air bath. The stove shall consist of (1) an air bath and (2) a top plate on which the flange of the cup rests

5.1.1 Air Bath – The air bath shall have a cylindrical interior and shall conform to the dimensional requirements in Fig F13. The air bath may be either a flame or electrically heated metal casting (Note 2), or an electric-resistance element (Note 3)

Note 2: If the heating element is a flame or electrically heated metal casting, it shall be so designed and used that the temperatures of the bottom and the walls are approx the same. The casting shall be designed so that products of combustion of the flame cannot pass up and come into contact with the cup

Note 3: If the air bath is of the electric-resistance heated type, it shall be constructed so that all parts of the interior surface are heated uniformly

5.1.2 Top Plate – The top plate shall be of metal, and shall be mounted with an air gap between it and the air bath. It may be attached to the air bath by means of three screws and spacing bushings. The bushings should have a thickness shown by dimension A and a diameter shown in dimension D in Fig F13

6. Thermometers

6.1 Two standard thermometers shall be used with the Pensky-Martens Tester, as shown in Table 1

Note 4: Whenever thermometers complying with ASTM requirements are not available, thermometers complying with the requirements for the Institute of Petroleum thermometer IP15F (or 15C) PM-Low, or IP16F (or 16C) PM-High, may be used, provided calibration corrections are used in temperature ranges where the IP requirements for scale accuracy are less stringent than those of ASTM

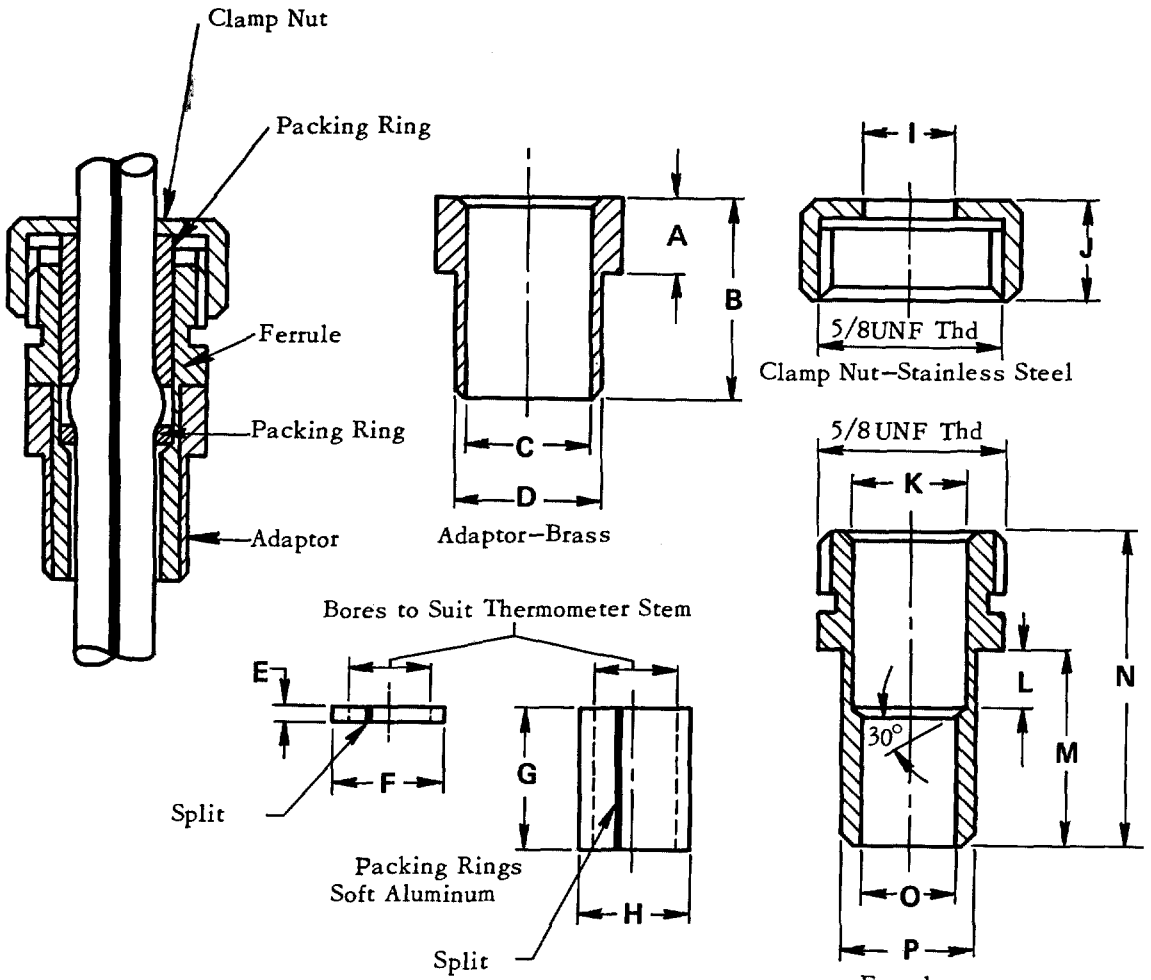


FIG A1 Thermometer, Adapter, Ferrule, and Packing Plug

	mm		inches	
	min	max	min	max
A	6.20	6.50	0.244	0.256
B	17.0	18.0	0.67	0.71
C	9.80	9.85	0.386	0.388
D	12.19	12.24	0.480	0.482
E	1.40	1.65	0.055	0.065
F	8.56	8.61	0.337	0.339
G	12.4	13.0	0.49	0.57
H	8.56	8.61	0.337	0.339
I	8.1	8.6	0.32	0.34
J	9.9	10.7	0.39	0.42
K	8.64	8.69	0.340	0.342
L	5.1	5.6	0.20	0.22
M	17.0	17.5	0.67	0.69
N	27.4	28.2	1.08	1.11
O	7.11	7.16	0.280	0.282
P	9.73	9.78	0.383	0.385

TABLE 1
Thermometers

Test Temperature Range	ASTM Thermometer
20 to 230 F	9 F (20 to 230 F) or 9 C (−5 to 110 C)
200 to 700 F	10 F (200 to 700 F), or 10 C (90 to 370 C)

APPENDIX A1. Manufacturing Standardization of Thermometer and Ferrule

A1.1 The low-range thermometer, which conforms also to the specifications of the cup thermometer in the Tag Closed Tester (ASTM Method D 56, Test for Flash Point by Tag Closed Tester) and which frequently is fitted with a metal ferrule intended to fit the collar on the lid of the Tag Flash Tester, can be supplemented by an adapter (Fig A1) to be used in the larger diam collar of the Pensky-Martens apparatus. Differences in dimensions of these collars, which do not affect test results, are a source of unnecessary trouble to manufacturers and suppliers of instruments, as well as to users

A1.2 Subcommittee 21 on Metalware Laboratory Apparatus, of Committee E-1 on Methods of Testing, has studied this problem and has established some dimensional requirements which are shown in Fig A1. Conformity to these requirements is not mandatory, but is desirable to users as well as suppliers of Pensky-Martens Testers

Flash Radiography of High-Speed Events. See X-Ray High-Speed Photography in Vol 2, p C17-R, under CAMERAS

Flash Reducer. In US Ordnance, any material issued separately for use with a propelling charge to reduce its muzzle flash. Differs from "flash suppressor" (qv)
Ref: Glossary of Ordn (1959), 119-R

Flash-Reducing (or Antiflash) Agents. Ordinary smokeless propellants, burning in the chamber of a gun at the expense of its own oxygen, produce a gas mixture containing N_2, CO, CO_2, H_2 and H_2O. The temp of combustion is usually so high that these gases may flash even before they leave the muzzle of the gun. This flash, however, is usually not as bright as that produced by the ignition of combustible components (such as CO and H_2) at the instant they come in contact with oxygen of the air at the gun muzzle. This after-ignition (known as **muzzle flash**) takes place close to the muzzle and is visible for long distances, especially at night. The larger the gun, the greater and brighter is the flash and the more difficult it is to eliminate (Ref 2, p 130)

As an example, accdg to Davis (Ref 5), when a 12 inch gun is fired there is a large and dazzlingly bright muzzle flash, which is about 150 ft in length. The light from such a flame is reflected from the sky at night and is visible for a distance as great as 30 miles, much farther than the sound from the gun travels. The enemy, by the use of suitable range finding apparatus, may determine the position of the flash and destroy the gun by long range bombardment. Other disadvantages of a bright flash from any gun, large or small, especially in night-fighting are obvious and, consequently, elimination of flash is of prime importance. The problem of "flash elimination" was originally investigated in France by Dautriche (Ref 1) and continued by Demougin (Ref 2), Fauveau & Le Paire (Ref 3) and Prettre (Ref 4). The subject has also been extensively investigated in the USA (Ref 5)

There are several ways in which the muzzle flash can be reduced or even eliminated
1) By incorporating in the smokeless proplnt substances such as KCl, NaCl, K_2SO_4 etc which volatilize and dissociate during the buming of the proplnt, thus lowering the overall temp of the combustn gases. It has also been found by Fauveau & Le Paire (Ref 3) and Prettre (Ref 4) that these substances will lower the ignition temp of CO in the muzzle gases when it comes in contact with air (See under "Antioxidants" in Vol 1, p A471). Accdg to Davis (Ref 7, p 325), potassium hydrogen tartrate also acts as an "antioxidant", but its action differs in that it decomposes and thus absorbs some heat from the gases

formed on deflagration of proplnt, thus lowering their temps. The same author (p 323) stated that tin or other substances which may be dispersed thruout the gas formed during deflgrn of proplnt also act as "antioxidants" or flash reducers

2) By incorporating in the proplnt organic nonexplosive substances, such as cellulose, vaseline, cellulose acetate, urea, hydrocellulose,, Centralites, stearic acid etc, which alter the compn of the gas mixture produced by the deflagrating proplnt by appreciably increasing the number of mols of gas formed, thus lowering the temp by "dilution"

3) By incorporating in the proplnt "cool explosives" such as AN, DNT, Mononitronaphthalene, Dinitroxylene, Guanidine nitrate, Nitroguanidine etc, which deflagrate with the production of gases which are considerably cooler than the gases from the deflgrn of ordinary smokeless proplnt. It should be noted that, when NG (Nitroglycerol) is replaced by NGc (Nitroglycol) in Double-Base and Triple-Base proplnts, "cooler" substances are obtained [See "Cooling Agents or Coolers" and "Cool (or Cooled) Propellants in Vol 3 of Encycl, pp C511-L to C512-L] (Double-Base and Triple-Base Propellants are described in Vol 5, pp D1536-L to D1539-L)

4) By incorporating in the proplnt substances such as $NaHCO_3$, $KHCO_3$, $(NH_4)_2C_2O_4$, $K_4Fe(CN)_6$, permanganates, chromates, oxalates, tartrates etc which decompose at high temps, absorbing some of the heat of combustion, thus lowering the overall temp of the gases

5) By incorporating in the proplnt small quantities of *Antiknock Compounds*, described as "Antidetonating or Antiknock Compounds" in Vol 1, pp A462-R & A463-L. One of such substances, tetraethyl lead, was proposed by the French. They were supposed to eliminate flash, at least partially (Ref 2, p 139)

6) By changing the design of the gun itself, or changing the design of igniter in such a manner that the projectile takes up energy from the proplnt gas more quickly and more efficiently, thus lowering the temp of the gases to a point where flash does not occur (Ref 7, p 324)

It is interesting to note that Black Powder was the original flash reducing agent. Dautriche added it in small quantities to Poudre B used in small arms and succeeded in eliminating flash completely. During WWI, the French successfully used flashless proplnts in machine gun shells which contained 90% Smokeless Propellant and 10% Black Powder. For large guns they used *anti-flash bags* ("sachet antilueurs") filled with potassium hydrogen tartrate or argols, which are by-products of the wine industry. The Germans used in their guns "antiflash bags" ("Vorlage") filled with coarsely pulverized potassium chloride or potassium sulfate which were loaded at the base of the projectile between the projectile and the proplnt charge

Accdg to Demougin (Ref 2, p 134) the best antiflash ("antilueurs"), arranged in approx order of decreasing effectiveness, are KCl (preferably paraffined to avoid the absorption of moisture), technical KH hydrate, KH oxalate, K nitrate alone or incorporated with 20% DNT, K sulfate and Black Powder (See also under "Cooling Agents", Vol 3 pp C511-L to C512-L)

Many of the materials used for flash reduction are also used in Permissible Explosives, suggesting that the two problems may be closely related

Following is a summary of proposed Flash-Reducing Agents, some of which have been used: K_2SO_4, KCl, KH tartrate, powdered Sn or SnO_2, NH_4NO_3, GuN, NGu, oxanilide, hydrocellulose, starch, anthracene, Nitroaromatic Compounds, NH_4 oxalate, camphor, triacetin, nitroquinolic acid salts, acetyl laurin, dibutyl tartrate, ethyl palmitate, TN-Naphthalene, phenanthrene, dicyandiamide, tricyantriamide, colophony, abietates ($C_{19}H_{39}COOH$) and cellulose (See also Ref 6)

Refs: 1) H. Dautriche, CR **146**, 535 (1908) 2) P. Demougin, MP **25**, 130–9 (1932–1933) 3) J. Fauveau & ?. LePaire, MP **25**, 142 (1932–1933) 4) M. Prettre, MP **25**, 160–69 (1932–1933) 5) C.G. Storm, Army Ordn **21**, 20–4 (1940) 6) Thorpe **4**, 521–23 (1940) 7) Davis (1943), 322–27

Addnl Refs:

A) W.O. Snelling, USP 1808613 (1931) & CA **25**, 4405 (1931) (Flashless proplnts obtd by

incorporating in Nitrostarch one of the following substances: GuN, AN, Urea Nitrate or NGu)

B) R.G. Woodbridge, USP 1838345; 1838347 (1931) & CA **26**, 1445(1932). See also CA **27**, 1177(1933) and **30**, 6946(1936)(Proposed addn of 0.5–2% K sulfate to proplnts)

C) R.G. Woodbridge, USP 1838346(1931) & CA **26**, 1445(1932)(Proposed stannous phthalate as flash reducer)

D) G.C. Hale & D.R. Cameron, USP 2026531 (1936) & CA **30**, 1233(1936); also USP 2035471(1936) & CA **30**, 3650(1936)(Flashless proplnt contg NC with 10–15% TNT and 2–10% triacetin or NC with triacetin alone. The triacetin functions as a flash reducing and gelatinizing agent)

E) E. Saigger, GerP 636977(1936) & CA **31**, 3280(1937)[Muzzle flash in firearms is reduced by placing at the base of the projectile one of the following: salts of nitrated quinol salts (such as K Dinitromethoxyquinol nitrate, $C_7H_{10}N_3O_8K$) or highly nitrated aromatic compds)

F) E.S. Goodyear, USP 2228309(1941) & CA **35**, 2722(1941)(Flashless nonhygroscopic proplnt: NC(N=13%) 73–79, DNT 21–24 and DPhA 0.8–1.2%)

G) T. Thomson & F.W. Whitworth, USP 2304037(1943) & CA **37**, 2938(1943) (NC proplnt contg 1–5% of an antimonyl compd free from halogens and contg a combined alkali metal, such as tartar emetic, which serves as an antiflash & antifouling ingredient)

H) G. Barsky, USP 2439291(1948) & CA **42**, 4349(1948). See "Flashless Propellant of Barsky"

I) G.C. Hale & L.F. Audrieth, USP 2480850 (1949) & CA **44**, 840(1950). See Flashless Gunpowder of Hale & Audrieth

J) J.N. Pring, USP 2557463(1951) & CA **45**, 9864(1951) [In cannon proplnts with NGu, substantial improvement in ballistic props results if its specific surface is controlled at a value in excess of 9000 sq cm/cc. Typical compn: NC(N=13.1 to 13.2%) 19, NGu(9000–22000 sq cm/cc) 55, NG 18.7 & DEDPhUrea 7.3%. The proplnt can be manufd by either conventional solvent or solventless process. Where flash reduction is particularly difficult 1 to 3% of K sulfate is helpful although the amt of smoke is increased]

K) A.M. Ball, USP 2577298 (1951) & CA **46**, 3764(1952) {Flashless mortar-type smokeless propellant in sheet form, such as: NC (13.25% N) 55.0, NG 33.65, cooler-plasticizer [such as o-$C_6H_4(CO_2Et)_2$]9.5, stabilizer DPhA 0.6, K nitrate (to aid ignition) 1.25% & desensitizer added methylcellulose 0.20 parts. Thickness of sheet controls ignition time}

L) J.T. Agnew, TransAmerSocMechEngrs **74**, 333–42(1952) & CA **46**, 4798(1952)(Line-reversal technique in detn of temp of gun flash is discussed. Monochromator is used to isolate Na, K, water-vapor radiation at 0.589, 0.77 & 0.942 microns resp. Peak temp values detd for external gaseous explns commonly known as secondary flash associated with firing a gun)

M) T. Okawa, JIndExplSocJapan **13**, 247–58 (1952) & CA **49**, 584(1955) (Muzzle flashes of TNT, BkPdr, Gelatin- and Ammonia-Dynamites expelled from mortar barrels were photographed by 16-mm camera at 4500–5000 frames per second. Jetting velocities of exploded gases were 1300–2300m/sec and decreased rapidly to 300–500m/sec. Max propagation vel of TNT's 2nd flame was 560m/sec

N) D.B. Murphy et al, PATR **2029**(1954)(High nitrogen compds as flash reducing agents)

O) B.W. Lewis & C. Boyars, USP 3097123 (1963) & CA **59**, 7311(1963) [Cool NC–base, non-C-forming proplnt, such as contg NC (12.6% N) 87.0, coolant (Ba stearate) 9.0, antiflash agent (K sulfate) 2.0, bore-lubricant (Basic Pb carbonate) 1.0 & EtCentr 1.0%. Calcd flame temp 1870cal/g and no C formed. Same compn w/o K sulfate produced C at 2000°]

P) F.A. Zihlman et al, USP 3116190(1963) & CA **60**, 7864(1964) [Proplnt with flame temp 1950–2200°K; there are no C deposits and reduced barrel erosion. Compn: NC(12% N) 87.5–91.4, EtCentr 2.8–6.0, lube (Basic Pb carbonate) 3.5–4.0, K sulfate 0.3 parts with some coolant (Ba stearate)]

Q) Anon, "Spectral Characteristics of Muzzle Flash", Army Materiel Command, **AMCP - 706–255**(1967)

Flash Reduction in German Projectiles. When it was required during WWII by the German High Command to have explosive flashes of Flak (AA) projectiles practically invisible in the night sky, the Krümmel Fabrik AG satisfied the requirement in the following manner: The high explosive filling was completely surrounded by a 5–6mm layer (sheath) of chlorine atom contg material, such as tetrachloro- or hexachloronaphthalene or by Amm chloride

Refs: 1) PBRept **925**(1945), Appendix 7
2) PATR **2510**(1958), p Ger 51-R

Flash Reduction in German Propellants (Mündungsfeuervermindung oder Mündungsfeuerdämpfung). In order to reduce the flash produced on combstn of proplnts, the Germans for many years used the salts of potassium, such as K sulfate, K nitrate, or K oxalate. The investigation conducted before WWII has shown that of the inorganic compounds the best flash reducers are the alkali salts and that flashlessness is improved on going up the series in the Periodic System. (Cs is better than Rb and Rb is better than K)

The inorganic flash reducers (such as K sulfate) were usually loaded in small bags separately from the proplnt, and placed between the projectile and the proplnt. These anti-flash bags, called in German "Vorlage", consisted of two perforated discs of artificial silk or cotton cloth sewn together in the form of "doughnuts" and filled with coarsely pulverized K sulfate. (Ref 1, p 324)

Another flash reducer consisted of a large bag with oxalic acid and a small bag with K oxalate

With the incorporation during WWII of Nitroguanidine (NGu) in some proplnts (See Gudolpulver), it was found that NGu alone gave sufficient flashlessness without incorporating any of the usual flash reducing agents. In proplnts which did not contain NGu, flashlessness could be successfully achieved by using a small bag with NGu and a small bag with K nitrate

It should be noted that the use of inert (non-explosive and non-combustible) flash reducers such as K sulfate, nitrate, or oxalate, oxalic acid etc, is always bound to decrease the ballistic potential of the proplnt and their use in large amounts should be avoided. This does not apply to NGu because this compd is not inert but is an explosivesive. For this reason, much larger amts of NGu may be used, either directly incorporated in a powder, or used in a separate bag

The following German flash reductants were examined at Picatinny Arsenal (Ref 3) during WWII:

a) K chloride; was used in 76.2mm AP weapons

b) K sulfate; was used in 7.92 Ball, 20mm APHV, 20mm Inc, 20mm HE Mauser, 20mm Solothurn, 37mm APHV, 37mm APMB, 37mm HE, 50mm HE, 75mm AP, 75mm HE and 100mm K18 weapons

c) Sodium bicarbonate; was used in some 88mm AP guns

d) Sodium sulfate; was used in some 75mm HE guns

According to Ref 4, the following compds were examined at the Düneberg Fabrik Dynamit AG as possible flash reducers **(Flammendämpfer):**

Aminoguanidine bicarbonate, Amm acetate, Amm phosphate, Amm sulfate, apatite, asbestos, Ba sulfate, boron nitride, cerium oxide, cryolite, dicyandiamide, dimethyl oxamide, dimethyl urea, disodium phosphate, mercurous nitrate, methylene urea, K bicarbonate, K chloride, K iodide, K metaphosphate, K perchlorate, K phosphate, K silico-fluoride, K urea oxalate, sodium ammonium sulfate, sulfur, zinc sulfate and Zr oxide

It was claimed that methylene urea reduced the flash to a far greater extent than any of the organic compounds used. It was also stated that cerium salts were much more effective than any other metallic salts investigated (Ref 4)

Abbreviations: **AP** Armor-piercing; **HE** High-explosive; **HV** Hyper velocity; **MB** Monoblock; **Inc** Incendiary

Refs: 1) Davis (1943), 324 2) O.W. Stick-

land et al, PB Rept **925**(1945), Appendix 8
3) W.R. Tomlinson, PATR **1555**(1945)
4) A.A. Swanson & D.D. Sager, CIOS Rept **29/24**(1946), p 6

Flash Signals. See Pyrotechnic devices and Flashlight Compositions used in flash signals

Flash Suppressor. In US Ordnance, any material incorporated into a proplnt to suppress flash. Differs from a "flash reducer" (qv) *Refs:* 1) Ohart(1946), 51 & 184 2) Glossary of Ordn(1959), 119-R

Flash Test for Caps. See Vol 1, p XIX, "Optical Method for Testing Caps"

Flash Tubes. Device used in mortar ammunition to transmit the flash from the primer element to the middle of the proplnt charge. Such an arrangement improved uniformity of ignition and reduced dispersion of fire. These tubes were formerly made from NC plastic which softened on absorbing NG from proplnt causing the tube to collapse. At present the tubes are manufd from a slightly impregnated rolled paper. See Vol **4**, pp D797-R to D803-L

Flat Plate Test and Cylinder Test. The *small scale plate test* is an experimental technique for evaluating the energy release from a detonating HE: It consists of a heavily confined column of HE, with a detonator & booster at one end, and a metal plate at the other end. The length of column is such that the results are independent of the detonator system. The test is run in a carefully standardized way with a constant vol of test expl; the measurement consists of determining the final plate vel by optical techniques. The plate is under acceleration for 2 to 4μsec (Ref 1)

A much larger experiment is conducted with the **flat-plate test.** Pin techniques are used to measure final plate velocity. For large scale testing of highly sensitive, expensive systems, the flat plate test has advantages over end-on tests in that lens & edge effects may be eliminated without using large amts of expl. The flat plate test also tends to accentuate the importance of the lower regions of the adiabat. The plate is under acceleration for a longer period (2 to 20μsec) compared to the small scale plate test (Ref 1)

Another test which shows considerable promise for exploring time-dependent adiabats with small amts of expl is the **cylinder test.** The std cylinder test geometry consists of a 1 inch diam, 12 inch long expl chge fitted into a Cu tube with a 0.1022 inch thick wall. A plane wave lens and 0.5 inch thick Comp B booster are used to initiate the test expl at one end. The radial motion of the cylinder wall is measured in a plane perpendicular to the cylinder axis 7 inches from the booster end. A streak camera records the motion, using conventional shadowgraph techniques. In addn, the deton vel of the expl is measured by placing pin switches 9 inches apart on the surface of the cylinder (Ref 2)

Some of the results obtd are as follows:

Energy Release Tests

Explosive	Density g/cc	Small-Scale Plate Vel mm/μsec	Flat Plate Velocity mm/μsec	Cylinder Test Velocity mm/μsec	Exptl Heat of Deton, H_2O liq kcal/g
Tetranitromethane (5 moles + Tetralin (1.5 mole)	1.47	1.16	1.58	1.43	1.56
Tetranitromethane (5 moles) + Tetralin & Decaborane (1 mole)	1.47	1.16	1.62	1.46	2.31
Composition B	1.72	1.08	1.76	1.55	1.22
PBX-9404	1.83	1.18	1.96	1.70	1.39

The above data show that the small scale plate test does not order expls in the same manner as larger more significant hydrodynamic tests

Some experimental cylinder test results at small and large expansions for a variety of expls are presented below:

Cylinder Test Results

Explosive	Composition	Density g/cc	Deton Vel mm/μsec	Cylinder Wall Velocity mm/μsec at $R-R_o$=5mm	Cylinder Wall Velocity mm/μsec at $R-R_o$=19mm
HMX	–	1.891	9.11	1.65	1.86
PBX 9409-03	HMX/NC/CEF 94/3/3	1.841	8.80	1.57	1.80
PETN	–	1.765	8.16	1.56	1.79
Octol	HMX/TNT 78/22	1.821	8.48	1.53	1.75
Cyclotol	RDX/TNT 77/23	1.754	8.25	1.46	1.70
Composition B	RDX/TNT 64/36	1.717	7.99	1.39	1.63
TNT	–	1.630	6.94	1.18	1.40
Nitromethane	–	1.143	6.37	1.01	1.22

The relative ability of these expls to deliver energy to metal has been confirmed in actual full-scale metal acceleration experiments

Refs: 1) J.W. Kury et al, 3rd ONR SympDeton (1960), Vol 3, pp 86–102 2) J.W. Kury et al, 4th ONR SympDeton (1965), pp 3–13

Flavanone or 2,3-Dihydroxyflavone, $C_{15}H_{12}O_2$, and other flavanones are reduction products of flavones and have similar occurrence and uses. Nitro derivatives with up to 3 nitro groups have been reported in the literature. They would appear of no expl interest having only 11% N

Refs: Same as under Flavone

Flavianic Acid. Same as 2,4-Dinitro-1-naphthol-7-sulfonic Acid, which will be described under Naphthol Sulfonic Acids

Flavone or 2-Phenylchromone, $C_{15}H_{10}O_2$, and other flavones are the parents of a number of yel natural dyes found in plants, leaves, fruits and flowers. The natural materials contain OH groups and occur uncombined or as glucosides. Nitration occurs in 2-phenyl group giving a mixture of 2'-, 3'-, and 4'-nitro flavones. If there is an OH group in the benzene ring, nitration may occur there. Compds with up to three NO_2 groups, but not higher, are reported in the literature. They would appear of no expl interest having only 11% N

Ref: E.H. Rodd, "Chemistry of Carbon Compounds", Elsevier Publg Co, NY, Vol 4(1959), 903–06

Flèche. Fr for Arrow

Flèchette. Fr for Small Arrow

Flechette (US) or **Aerial Dart.** A small *fin-stabilized missile* (See "Fin Stabilization and Spin Stabilization" in this Vol), a large number of which can be loaded in an artillery canister consisting of a light sheet metal casing, designed to be fired from a rifled gun. The flechette projectile, shown here as Fig F17, was developed in the USA in the early 1960's. It has a very high length/diameter ratio. Because it will be supersonic in flight, the *fins* (four of them) must stand out well clear of the body to be in the Mach wave induced by the nose. To enable the maximum propelling force to be developed, a *sabot* (which is of plastic) was required to produce a larger cross-sectional area. Since the proplnt space is limited and must be utilized to the fullest, the sabot must be a puller, rather than the conventional pusher type associated with "armor-piercing discarded sabot" (APDS) rounds used in large-caliber tank and antitank guns. Also, since the flechette is not spun, there is no centrifugal force available to dislodge the sabot at the muzzle, as there is with APDS. This means that a mechanical cutter is required to effect the separation of the sabot from the flechette. This, in turn, adds to the complexity of the system and greatly increases the possibility of inaccuracy

Assuming that the launch of the projectile is made without imposing initial errors on the flight path, it then will proceed along the trajectory with the unspun body stabilized only by the fins

The aim of Major Hobart's paper (Ref 2) was to examine the parameters affecting spun and fin-stabilized flight with a view to deciding which provides the better system for a low-impulse, lightweight, hand-held military weapon of small caliber. Typical examples

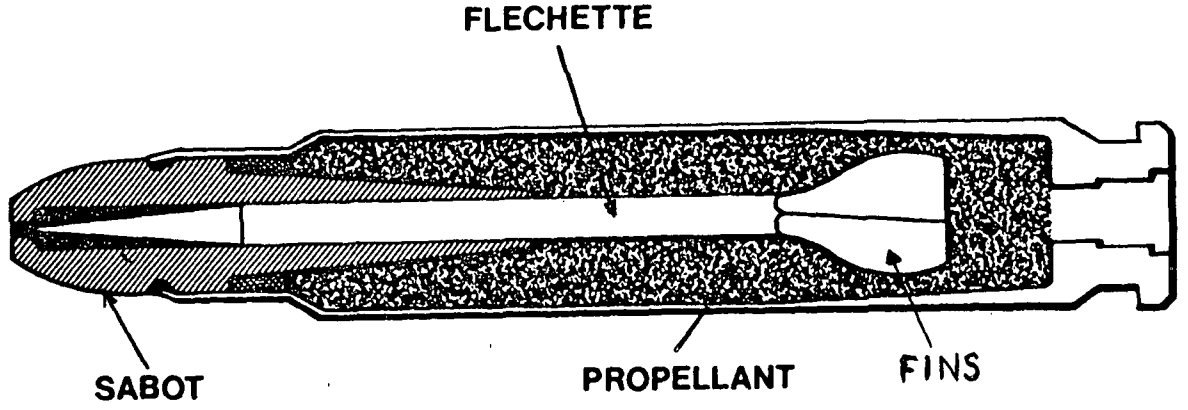


FIG F17

would be AR 15 caliber .223 rifle and the "Special Purpose Individual Weapon" (SPIW). The SPIW was briefly described in Ref 1. It is a rifle firing flechette, which is approx an inch long and approx as thick as the lead in a pencil

The following discussion is that of Major Hobart, as given on pp 314 & 315 of Ref 2:

The spinning of a projectile is produced by the engraving of the bullet envelope on the grooves of the rifling cut into the bore of the barrel. The rate of rotation depends on the steepness of the rifling spiral (usually expressed as one turn in x inches; it may be referred to, in larger guns, as one turn in x calibers, or the helix angle may be the reference parameter)

The twist is almost universally constant in pitch, although older weapons with an increasing twist may be encountered occasionally. In the latter case – provided the rifling continues to grip the envelope thruout the bullet travel in the bore – the final angle of twist decides the rate of rotation

In a modern military rifle, one turn in 12 inches is a very common twist, and this enables an immediate mental calculation of the rate of revolution of the projectile at the muzzle – since for every one foot the bullet travels forward in a second, it rotates once. So, with a muzzle velocity of 3250ft/sec, the bullet is spun at 3250 revolutions/sec

For other degrees of twist, the rate of revolution can easily be calculated. For example, at a twist of one turn in 10 inches, the rate is 12/10 x 3250/1revs/sec; at one turn in 14 inches, the rate is 12/14 x 3250 revs/sec

The degree of twist in a modern rifle is generally just about the minimum that will produce stability in a bullet passing thru an atmosphere of a density corresponding to a temperature of zero degrees centigrade. This is to ensure that entry into a denser medium – i.e. the target – will produce complete instability, which is a desired criterion for wound ballistics

When a bullet is fired at short ranges, it is generally just stable in its flight thru the air. When fired at long ranges, the forward velocity is lost at a greater rate than its angular velocity, and it becomes "overspun". This results in the bullet's failing to remain in flight with its longitudinal axis along the trajectory; it adopts a nose-up attitude producing an angle with the trajectory (See Figure 1). Its behavior, then, is exactly that of a gyroscope

If the bullet is spun clockwise by the rifling, the wind force tending to push the nose up induces a precession, and the nose of the bullet is turned to the right of the trajectory as viewed from the gun. This in turn produces a further precession, and the nose rises

Thus, the effect of the overspun characteristic is to cause the bullet to drift bodily to the right, away from its original line. If there is a strong cross wind, the bullet will be displaced in the direction of the air flow

A bullet from a military .223 rifle launched at 3250ft/sec will lose its velocity quite rapidly until, at 400 meters, its residual velocity will be 1800ft/sec and its remaining kinetic energy ($\frac{1}{2}$ mv^2) will be $\frac{1}{2}$ 55/7000 x $1800^2/g$ ft lbf or $2.2 \times 10^{-6} \times 5.5 \times 1.8^2 \times 10^7$; i.e. 395 ft lbf (See Figure 3)

Since the accepted figure for the production of an incapacitating wound on an unprotected man is 58 ft lbf, it will be seen that, if all this energy can be conveyed to the target, it will be more than enough

The bullet, on striking the target which is denser than air, immediately becomes unstable and yields up its energy to the target. Thus the spun bullet of the characteristics described is perfectly adequate for unprotected targets out to a range of 400 meters

A fin-stabilized projectile must have its center of mass well forward of its center of pressure. This means that the point thru which gravity can be said to act must be well in advance of the point at which the aerodynamic forces act. A good example of this is a metal dart which has a brass body of considerable density and fins placed on a lightweight wooden boom well to the rear

When a fin-stabilized projectile is *yawing*, the wind pressure exerted at the

center of pressure will rotate the rear end of the projectile about the center of mass, until the air flow exerts equal pressures on each side of all the fins, thus placing the longitudinal axis directly into the air flow. This *weather-cocking* effect is most pronounced when the distance of the center of pressure behind the center of mass is large, and the magnitude of the restoring moment is at its greatest (See Figure 2)

This system works well in normal flight, but – at the moment of emerging from the muzzle – the gases following up along the bore expand, accelerate, and rush past the projectile which is, therefore, at that moment flying backward relative to the air flow. This so-called intermediate zone covers the first 6 ft or so of travel from the muzzle. The effect of this reversed air flow is to increase any initial yaw at the muzzle by a factor which may be as high as 10. Thus it is absolutely imperative that initial launch conditions be perfect so that there is no yaw

As the missile proceeds along its trajectory – assumed to be low-angle fire – there is no tendency for it to depart therefrom, and the longitudinal axis will be restrained by aerodynamic forces to follow the trajectory

To get the center of mass well forward in a flechette of the shape shown in Figure 2, it is obviously necessary to have a long body. Reference to the title illustration shows a typical flechette with a length of 1 inch, a diameter of 1/10 inch, and a weight of 10 grains. Due to this low mass it cannot travel far, because it loses velocity very quickly

A side wind will cause the fin-stabilized missile to turn into the wind. Therefore wind correction, when aiming, will be the opposite of that applied for a spun projectile

The flechette, due to its light weight, can achieve a very high muzzle velocity – of the order of 4500ft/sec. It loses velocity quickly, but at the maximum range at which a rifle can be expected to be used in war, i.e. 400 meters – it still is travelling at about 3000ft/sec. Its kinetic energy is then $\frac{1}{2}$ 10/7000 x $3000^2/g$ ft lbf = $2.22 \times 10^{-6} \times 9 \times 10^7$ = 199.8 ft lbf, which is about 3½ times that considered necessary to produce an incapacitating wound (See Figure 3)

Due to its long, thin shape, there is a lack of rigidity, and the flechette turns into a hook on impact with the target. It becomes totally unstable and imparts its full kinetic energy to the target, producing an explosive-type wound

For a long-range weapon, therefore, the greater mass of the spun projectile is superior to the flechette

For ranges up to 400 meters, the target effect is much the same, but the accuracy

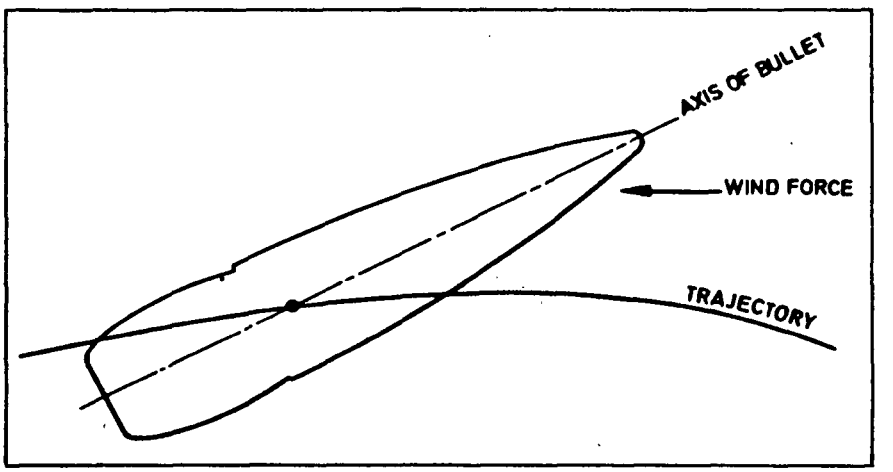


Fig 1 Diagram of bullet rigidity and its operation during flight

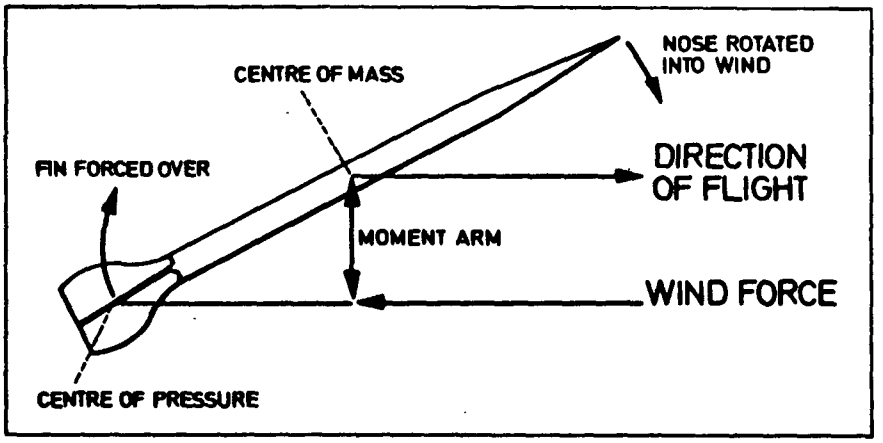


Fig 2 How the "weathercocking effect" influences the flight of a flechette

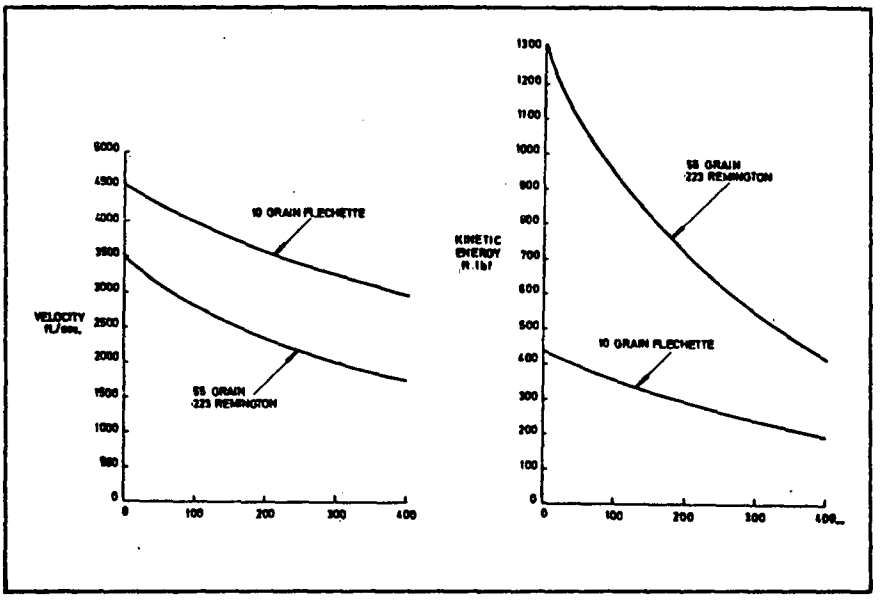


Fig 3 Comparisons between a 10-grain flechette and a 55-grain Remington caliber .223 bullet, regarding remaining velocity (left graph) and kinetic energy per ft lbf (right graph), both vs range

of the flechette depends entirely on launch conditions. If the projectile can emerge from the muzzle without yaw, its accuracy is as good as that of the bullet and its higher velocity, leading to a lower vertex height, will reduce range-finding errors and increase the chance of a hit. The shorter time of flight will reduce the lead required on a moving target

At short ranges, the effect of a cross wind is about the same in both cases – but in opposite directions

There would seem to be a considerable future for the flechette used in a low-impulse, lightweight military weapon, provided its launching conditions at the muzzle can ensure no initial yaw

Refs for Flechette: 1) Anon, "New Infantry Weapon", Ordn **48**, 405 (1964) (A brief description of SPIW) 2) Major F.W.A. Hobart, "Fin Stabilization or Spin", Ordn **57**, 313–15, Jan-Feb 1973

Flegmatizatory. Rus for **Phlegmatizers** (qv). Accdg to Shilling (Ref 1) and Blinov (Ref 2), substances such as oil, paraffin, vaseline, wax, etc have been used to desensitize expls and also for diminishing the erosive action of propellant gases. (See under EROSION OF GUN BARREL, in Vol **5**, pp E112-R to E120-R)
Refs: 1) Shilling (1946), 167 (in Rus)
2) Blinov, "Kurs Artillerii", Vol **5**(1949), 163
3) PATR **2145**(1955), p Rus 6

Flegmatizirovannyi Gheksoghen (Phlegmatized RDX). A mixt of RDX with a small quantity of wax paraffin or ceresin pressed to a d 1.65; used as base charge of detonators
Ref: Gorst (1957), 100 & NewEdn (1972), 137

Flegmatizirovannyi TEN (Phlegmatized PETN). A mixt of PETN with up to 5% wax or paraffin has been used for loading shaped charges and other shells as well as in detonators
Ref: Gorst (1957), 95 & NewEdn (1972), 132

Flegmatizirovannyi Trotil (Phlegmatized TNT). A mixt of TNT 94, naphthalene 4 and DNB 2% used during WWII for loading AP shells (broneboynykh sn ariadov)
Ref: Gorst (1957), 98 & NewEdn (1972), 135

Flerlet Powder (1870). K chlorate 40, sugar 20 & sulfur 20%
Ref: Giua, Trattato **6**(1)(1959), 392-R

Flexed TNT. The design of continuous (lined) TNT charges for *Mark 29 Streamliners*, used in Mark 29 Antitorpedo Device. Studies at the Explosives Research Laboratory, Bruceton, Pa, have led to the development of the "Flexed TNT" charges. The method of prepn consisted in filling a reinforced rubber hose with molten TNT and subjecting the hose, while the TNT is cooling and solidifying, to a mild flexing operation. Several designs of flexing machines were described, the last of which proved to be satisfactory in rather extensive operations undertaken at the Yorktown Naval Weapons Station, Yorktown, Virginia. The properties of the Flexed TNT chges, which consisted of fine-grained material, were as follows: chge density 1.4g/cu cm, length of propagation unlimited for hose as low as 1 inch diam, unaffected by moisture below 12% and insensitive to rifle bullets

Charges of diams from one to 2½ inches were successfully prepd. They were formed in reinforced rubber hose of wall thicknesses varying from 1/16 to 1/4 inch (Ref 1)

The principle embodied in Mark 29 Antitorpedo Device was as follows: Two explosive streamers and a third streamer contg audio pickups sensitive to vibrations from the torpedo were to be trailed by a faired (joined so that external surfaces blend smoothly) cable attached to a *paravane* (See Note). When a torpedo passed over or under the detector streamer, one of the explosive streamers would be fired automatically to destroy the torpedo. The 2nd streamer would then be thrown automatically into the firing circle in readiness for a 2nd attack (Ref 2)
Note: Paravane is a device, usually torpedo-shaped, for protecting a ship from moored mines. It is towed by a cable and has a device that cuts the mine cable, bringing the mine to the surface. Paravanes are always used in pairs, one towed from each bow (Ref 3)

The first expl streamer developed for the Mark 29 device by the Eastern Laboratory (EL) of the duPont Co, Gibbstown, NJ was made with pressed TNT pellets or blocks. The hose couplings, buoyancy control (air pressure) and method of initiation (by Primacord) were the same as for the NOL Streamer in use at that time. The only apparent weakness of the duPont streamer was that the pellets were broken to some extent by handling and counter mining. The disadvantage of the pellet load was largely eliminated by the EL development of a blend of flaked and grained TNT for use in the NOL canvas sock

A series of water-compatible expls based on Pentolite was developed, the preferred type being a slurry of Pentolite, AN & water with/or wo Al powder. These slurries could be poured rapidly into hose of 1¼ inches or greater diam and of length 200 ft or more. For

smaller diam hose loading was by air-pressure extrusion device (Ref 2)
Refs: 1) J.H. Lum & J.K. Bragg, "The Design of Explosive Streamers", OSRD **1382**, May 1943, abstracted in OSRD **6630**(1946), pp 62–3 2) Eastern Laboratory, Explosives Department, E.I. duPont de Nemours and Company, "Development of Linear Charges: Final Report on Mark 29 Device", OSRD **3781**, June 1944, abstracted in OSRD **6630**(1946), pp 146–48 3) "The American College Dictionary", Random House, NY(1952), 880-L

Flexible Explosives. "Detasheet" explosives, consisting of PETN, RDX or HMX and elastomer binder in cord, bar or sheet form, are described in new, updated booklet. Changes include revised sections, addition of "Detasheet" D, and inclusion of federal stock numbers. *Detasheet Flexible Explosives, Booklet A-62232, E.I. duPont, Pompton Lakes, NJ 07442.* (See also Vol **3** of Encycl, pp D99-L to D101-L)

Flex Test for Barrier Materials. A mechanical flex test to replace the US Specification MIL-B-131B Performance Test of water vaporproof barrier material (July 1952), which is costly and time-consuming, was developed at Picatinny Arsenal. Three mechanical flexing devices, each having a basically different action, were evaluated as possible substitutes for the above spec test:

a) MIL-B-131B Gelbo Tester (modified)
b) Delamination Tester flexing at 160°F/100% RH and under ambient conditions
c) Point-Flex Tester

The point-flex test developed in this work was not considered an adequate substitute for the MIL-B-131B Performance Test. This test, like the Water Vapor Transmission Rate (WVTR) test after low-temp flexing at −20°F separates good from poor materials in the same manner as the spec test, but does not rate barrier materials in the same order of quality

It was recommended, however, that Specification MIL-B-131B be revised and some features of the point-flex test and max WVTR values for barrier materials be incorporated
Ref: R.H. Evans & S. Liff, "A Flex Test for Watervaporproof Barrier Materials", PATR **2302**(June 1956)

FLEX-X. An adhesive-backed version of *Detasheet* (See Vol **3**, pp D99-L to D101-L)

Flexible Hollow Spheres. Lethal hollow spheres which may be used to destroy enemy targets in outer space by high velocity impact are patented by Hershkowitz (Ref). The hollow spheres are formed of resilient material which is highly impregnated with metallic particles. The spheres are flattened, partially evacuated and sealed so that each contains a small predetermined vol of gas or air trapped therein at STP. Thus, they occupy less space and a greater number of flattened spheres can be packed in a rocket or shell for subsequent projection toward a selected target in outer space. As the spheres are projected thru space or rarefied air toward a target, the relative increase in pressure of the expanding gas or air trapped in the spheres causes them to expand outwardly to their full spherical shape when discharged from their delivery container for lethal impact
Ref: J. Hershkowitz, USP 3749016(July 1973)

Flintlock or Firelock. A small-arms firing device ("gunlock") invented in Spain in the middle of the 17th century to replace the earlier device, known as *wheel-lock* (See Ref 3, p D754-L & R). The early version of flintlock was known as *snaphance* (See Ref 3, p D755-L). The device was improved in 1670–1680 to such an extent that it started to be used by all nations. In the latest model, the flint was clamped in a spring-driven cock, controlled by a sear (a pivoted latch) operated by the trigger. When the cock fell under the action of the spring, the flint struck a steel hammer, which was hinged over the flash pan, protected by a cover from rain or fog. The blow uncovered the pan and produced a shower of sparks directed into the priming powder. The improved version of flintlock was used by the British until about 1850 and by the Americans as late as the end of the Civil

War. It was replaced by *percussion-lock*
Refs: 1) M.B. Garber, "A Modern Military Dictionary", Washington, DC (1936), 135 2) T.J. Hayes, "Elements of Ordnance", Wiley, NY (1938), 625 3) Fedoroff & Sheffield, Encycl of Expls, Vol 4(1969), pp D753-R to D756-L (Brief description of match-lock, wheel-lock, flintlock and percussion-lock in Section entitled "History of Development of Military Detonators, Igniters, Primers and Other Initiating Devices")

Flobert Rifle and Ammunition, known in Russia and some other countries as *Monte-cristo,* is briefly described in Ref 4, p D756-L. Although invented in 1840, it is still used for target practice and shooting small birds. The ammunition consists of a short metallic cart-ridge, cal .22, (resembling a large blasting cap), charged with a primary mixture: $MF + KClO_3 + Sb_2S_3$, (serving as a proplnt) and a small lead bullet. The ignition of mixt is caused by the compression of the rim of the case against the breech of the barrel when it is struck by the hammer of the firing mechanism. The pressure of gas developed in the cartridge is sufficient to propel the bullet a few yards

In order to be able to fire at longer range, the Amer inventors H. Smith & D. Wesson made the cartridges longer and placed a charge of BkPdr betw the primary mixt and bullet. The early models of Smith-Wesson revolvers had these cartridges

The same system was adopted in larger than cal .22 weapons, including cal .41 *Vetterli Repeating Rifle* used in 1870's & 1880's by the Swiss army

Rim fire weapons (such as Flobert) are replaced by *center-fire* weapons (See Ref 3, pp C74 & C75)
Refs: 1) A. Marshall, "Dictionary of Explosives", Churchill, London (1920), 40 2) M.M. Johnson & C.T. Haven, "Ammunition, Its History, Development and Use, 1600–1943", W. Morrow, NY (1943), 38–43 3) Fedoroff & Sheffield, Encycl of Expls, Vol 2(1962), pp C74 & C75 4) Ibid, Vol 4(1969), p D756-L

Flocculation. The formation of larger particles of a solid phase dispersed in a solution by the gathering together of smaller particles. The process whereby initial aggregates (having dimensions of a few unit cells) in a soln develop spontaneously into particles of a new stable phase is known as *nucleation.* When these particles grow to size sufficient to scatter visible light, they are known as *colloids.* Dispersions of particles of colloidal size (colloidal solns) are frequently stable, coalescence of the particles into larger aggregates settling under gravitational or centrifugal forces being prevented by their similar residual and mutually repelling chges. By changing the ionic environment in which colloidal particles exist, for example, by adding salts contg multi charged ions, the colloidal particles can be made to undergo further aggregation or flocculation. Silver chloride is a good example of a compound which can be precipitated as a flocculated colloid
Refs: 1) L. Gordon & R.W. Murray, "McGraw Hill Encyclopedia of Science and Technology", McGraw Hill, NY, Vol 5(1971), p 33

Flogel. A series of metallized expls, consisting of slurries of finely divided Al–AN or Al–AN–TNT have been manufd since 1961 at the Hercules plant in Gilbert, Minn and since 1963 in Ishpheming, Mich
Ref: Anon, C&EN **42**, 30-L, 27 April, 1964

Flotation or Froth Flotation. According to De Vaney (Ref, Perry, p 1088) *flotation* is a "process whereby the grains of one or more minerals, or chemical compound in a pulp or slurry, are selectively caused to rise to the surface in a cell or tank by the action of bubbles of air. The grains are caught in a froth formed on the surface of the tank and are removed with the froth, while the grains that do not rise, remain in the slurry and are drawn off the bottom of the cell or tank

Floatability is a surface phenomenon and some solids are more easily floatable (such as sulfur, sulfides of the metals, graphite) than others (such as oxides, silicates, silica).

Agents used to form a froth are pine oil, cresylic acid and the branched chain alcohols sold by duPont as B series or a mixture of these, alcohols with fuel, or pine oil, sold by the American Cyanamid Co as "AC series"

The first oil flotation plant was installed about 1914 and since then it has replaced to a certain extent the "Tabling Method" (See Note, below) developed in about 1896 by Wilfey. Tabling is still used for the treatment of material that contains only one valuable mineral of granular size and where a considerable difference exists between the effective specific gravities of the mineral constituents. Flotation, on the contrary, is best suited for treating complex ores containing several valuable minerals, those requiring fine grinding for liberation and those having small gravity differentials

NOTE: Tabling is a concentration process whereby a separation between two or more minerals is effected by flowing a pulp across a riffled plane surface inclined sl from the horizontal, differentially shaken in the direction of the long axis and washed with an even flow of water at right angles to the direction of motion

Dry Tabling has a shaking motion somewhat similar to that of a wet table, except that the direction of motion is inclined upward from the horizontal, and instead of water acting as the medium of distribution, a blast of air is driven thru a perforated deck

Agglomeration Tabling is a process whereby selective flocculation or agglomeration of grains of one mineral in an aggregate is caused by the addn of an agglomerating agent in a conditioning cell or in the ball-mill circuit, the slurry contg the aggregate grains then being fed across gravity tables (Ref Perry, 4th edit (1962), pp 21-61 to 21-63

Refs: 1) A.M. Gaudin, "Flotation", McGraw-Hill, NY (1932) 2) A.M. Gaudin & P. Malozemoff, TransAmInstMiningMetEngrs **112**, 303–18 (1934) 3) W. Petersen, "Schwimmaufbereitung", Steinkopf, Leipzig (1936) 4) A.M. Gaudin, "Principles of Mineral Dressing", McGraw-Hill, NY (1939) 5) A.F. Taggart, "Handbook of Mineral Dressing", J. Wiley, NY (1945) 6) Flotation Index, A Bibliography of Articles on Flotation (published by Great Western Div, The Dow Chemical Co, San Francisco, Calif (1946) 7) F.D. de Vaney et al, IEC **38**, 20–21, 35 (1946) (Review on flotation with 17 refs) and in succeeding years 8) H. Hagihara, "Surface Phenomena in Flotation", Mitsubishi Mining and Metallurgical Laboratory, Japan (1950) 9) J.H. Perry, "Chemical Engineers' Handbook", McGraw-Hill, NY (1950), pp 1085–1091; F.D. deVaney, "Froth Flotation" (Numerous refs) 10) R.E. Kirk & D.F. Othmer, Edit, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **6** (1951), p 595–614; H.R. Spedden, "Flotation"; with 22 refs 11) K.L. Sutherlands & I.W. Wark, "Principles of Flotation", Australian Inst of Min & Met, Melbourne (1955) 12) Perry, 4th edit (1963), pp 21-70 to 21-75 13) Kirk & Othmer, 2nd edit **9** (1966), pp 380–98; F.F. Aplan, "Flotation"- with 67 refs

Flo-Turn Process for Forming Shell Bodies. Picatinny Arsenal now includes among its new precision and cam controlled manufg equipment a flo-turn machine capable of manufg contoured or tapered shells up to 42 inches in diam and 60 inches long. Flo-turning is basically a cold rolling process in which the displacement of metal is parallel to the centerline of the part being formed. This movement of metal is produced by action of a cam controlled & hydraulically operated roller against a blank to be formed which in turn is backed up by a mandrel

Metal displacement in the flo-turn process differs from metal displacement in the cold rolling mill-process in that flo-turning displaces the metal in a spiral manner as work revolves as compared to the displacement of metal in a longitudinal direction in the cold rolling process

Flo-turning differs from spinning in that metal is displaced rather than bent into shape. The wall thickness of a spun part is basically maintained but the outside diam of the blank is reduced as it is wrapped over a mandrel. Flo-turning maintains its outside diam thruout the process and has a resultant wall thickness equal to the sine of the angle of the finished piece multiplied by the blank thickness

The advantages of producing parts by the flo-turn process are as follows:

a) seamless parts may be produced

b) tolerances comparable to those achieved by machining are possible

c) surface finish is of high quality as the metal is burnished rather than cut

d) complex contours may be flow turned

e) metal waste is almost eliminated

f) mechanical props of the material may be greatly improved due to extreme cold working

g) in-process heat treatment may be eliminated by using work hardening alloys

h) when welding preforms are used, joints are improved by plastic deformation and are less noticeable on the finished part

i) unit cost is low due to speed of the process

j) tooling cost is moderate allowing economical production of a relatively small number of parts and

k) integral circumferential stiffening members may be incorporated at the ends of the parts

A major accomplishment of the flo-turn process is the manuf of the Davy Crockett Projectile Body

Ref: Anon, "Picatinny Arsenal's Capability for Manufacturing Contoured Skin Shells by Flo-Turn Process', Methods Engrg Branch, Arsenal Operations Div Rept **ME-4-61** (Oct 1961)

Flour. Finely ground grains of wheat, rye, corn, etc consisting of starch, gluten, etc. Besides its use as food, flour is used as combustible (carbonaceous) ingredient of expl compns, such as Gelignites, Gomme E, Dynamite II, Belgian Explosifs SGP, German Kohlen-Carbonit, etc

Ref: Marshall 1(1917), 172, 273 & 276
2) Davis (1943), 340–51

Flour, Cellulosic. To this belong sawdust, woodflour, woodmeal, woodpulp, etc. For example, woodmeal has been used in French expl combns, such as Dynamite-gommes and Gélatines B (potasse) and B (soude); Belgian Forcites used to be manufd at Baelen-sur-Nèthe; British Saxonite, Samsonite, Arkit No 2, Duxite, Cambrite, Britanite No 2, Super-Kolax No 1 & No 2; and German Carbonit, Tutol & Kolax

Ref: Marshall 1(1917), 372–76

Flour Tester. An apparatus invented in Sweden for approx measurement of particle size of wood pulps. It consists of various sizes of sieves and a device to feed water at a constant rate and at constant pressure of ca 2kg/sq cm. A slurry contg about 2g of woodpulp (on a dry basis) per liter of water is placed on a sieve which has the required openings. Water is admitted at constant rate & pressure and, after running from 3 to 5 mins, is then closed and the sieve removed, dried & weighed. The percent pulp retained & passed thru the sieve is determined. Repeating the operation using smaller sizes of sieves, Brissaud used this app to determine the fineness of various Nitrocelluloses and found the method to be simple & reliable

Ref: L. Brissaud, MP **30**, 201–04 (1948) (Mesure de la finesse des cotons-poudre. Examen d'un appariel suédois "Flour Tester")

Flow of Fluids (Flow of Liquids and Gases). See under Fluid Mechanics or Dynamics

Flow Laws of Detonation. See J.F. Roth, Explosi vst **1958**, 23–31 & 45–54, "Die elementare Ableitung der Strömungsgesetze der Detonation" (The elementary derivation of the flow laws of detonation). See also Detonation, Chapman-Jouguet Wave and Flow Behind It in Vol **4**, pp D236-L to D237-L

Flow Velocity in Shock. See Vol **4**, p D516-R, Ref 11 and Fig 5 on p D517-L

Flow Velocity of the Detonation Products of Explosives. Formulas, based on hydrodynamic theory, were developed for the detn of the detonation products of gaseous mixtures and

condensed explosives such as TNT, PA and Tetryl
Refs: 1) L.D. Landau & K.P. Stanyukovich, CR AcadSci(Russia) **47**, No 3, 199–201(1945) 2) Ibid, No 4, 271–4(1945) & CA **40**, 4217 (1946)

Flox. Fluorine/Oxygen Mixture for Atlas Rockets. A research program of the Lewis Research Center of NASA using this new propellant indicates that the pay load capacity is increased by 90% for 160km high orbits and by 65% for tasks with escape velocity. About 30% fluorine & 70% oxygen is taken as a basic mixt
Ref: Dr A. Langhans, Explosivst **12**, 196(1964)

Fluctuating Velocity in Detonation Wave. See Detonation Wave with Fluctuating Velocity in Vol **4**, p D728-L, Ref 66, p 173, Item B

Flueric Explosive Initiator. See V.P. Marchese, Singer Company Final Rept **KD 72–75** (Nov 1970 to July 1972)

Fluerics. See Fluidics for Ordnance in this Vol

Flueric Safety and Arming of Selected Ammunition. See V.P. Marchese, Singer Company Final Rept **KD 72–68** (June 1971 to July 1972)

Flugkörper (Ger). Missile

Flugzeugträger (Ger). Aircraft Carrier

Fluid Actuator. A new actuator, called *PyrAc*, is a low cost fluid dispenser or fluid power supply powered by a pyrotechnic charge. It is non-fragmenting and is hermetically sealed. The device consists of two concentric tubes sealed at the end with a pyrotechnic charge in the inside of the fluted inner tube. The space between the fluted section and the outer cylinder provides the reservoir for the hydraulic fluid. When the inner tube is pressurized by the pyrotechnic, it inflates to cylindrical shape expelling the stored fluid or gas. The unique construction of the actuator permits long term storage of a sealed fluid reservoir because thermal expansion of the fluid is accomodated by flexing of the fluted portion of the inner tube. Applications are said to include fluid power supply for missiles, gas pressure for bag inflation for auto crash safety, and light gas gun propulsion
Refs: 1) Olsen Development Lab, USP 3483695(1969) 2) Expls&Pyrots **3**(6)(1970)

"Fluid Ball". Trademark of Olin Mathieson Chem Corp, Stamford, Conn 06904, for a proplnt casting powder consisting of fully colloided NC having an average particle diam of 50 microns or less. Compn can include liq & solid modifiers. Used as binder constituent of modified double-base rocket proplnts
Ref: CondChemDict(1961), 503-R & 504-L; 8th edit(1971), p 394-R

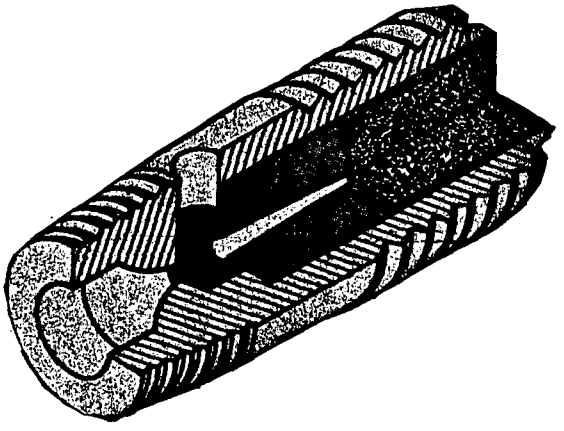
Fluid Explosives, Reactions Following Initiation of. Initiation by gas compression and heat in various confinements is discussed for TNT, NG and German mining expls
Ref: M. Zippermayr, Explosivst **3/4**, 25–40(1955) & CA **49**, 8601(1955)

Fluid Flow. See under Fluid Mechanics or Dynamics and the following Refs
Refs: 1) R.F. Steams et al, "Flow Measurements eith Orifice Meters", VanNostrand, NY (1951), 384pp 2) J.R. Caddell, "Fluid Flow in Practice, "Reinhold, NY(1956), 125pp 3) Perry, 4th edit(1963), pp 5-5 to 5-58(Flow Measurement and Fluid Dynamics)

Fluidic Explosive Initiator. A new fluidic explosive initiator operates on compressed gas and utilizes acoustic resonance instead of electricity to initiate an explosive charge. Current applications are in various military

weapons systems and for solid rocket and sounding rocket ignitors. Because of its safety features (unique all-fire signal and absence of electrical path thru the unit) manufacturer also recommends it for use in commercial applications, including as a squib switch, replacement for standard electrical detonators in mining and heavy construction, as a safety valve in nuclear power plants and in aircraft and industrial applications where emergency procedures depend on electrically powered squibs which, in an actual emergency, would probably not have the required energy available

Operating on gas pressures of approx 40 psig, the initiator will ignite primary explosives or propellants in 5 to 10 milliseconds. A predetermined time delay can also be built into the device. In testing for NASA Langley, ignitions were obtained



at stand-off distances of 330 ft. Features of the initiator are said to include safety, simplicity, reliability, long storage life and low cost. The initiator consists of two parts, a resonance tube and an excitation nozzle. In operation, gas flow is converted directly into thermal energy which is used in the initiation process. While the resonance tube theory for the generation of thermal energy is not new, its application in fluidic systems is unique. The device is 0.825 inch long, 0.250 inch in diam and weighs 0.125 ounce

Refs: 1) EMX Engineering, Inc, 354 Newark-Pompton Turnpike, Wayne, NJ 07470
2) G. Cohn, Edit, Expls&Pyrots **7**(4), (1974)

Fluidics for Ordnance. *Fluidics* is the general field of fluid devices and systems with their associated peripheral equipment used to perform sensing, logic, amplification and control functions. *Fluerics* is that area within the field of fluidics in which fluid components and systems *perform* sensing, logic amplification, or control functions, without the use of moving parts. The application of fluidic techniques to ordnance has been mainly in the field of fuze arming systems. More recently, attempts have been made to apply these devices to fuze design (Refs 3 & 6). In general, fluid operated devices can be used to transfer motion with an amplified force or displacement, provide arming or functioning delays, and program events for complex devices. The field of fluid mechanics is large and complex but well covered in standard texts (Refs 1 & 2)

Present technology predicts that many of the control and sensing functions can be accomplished by flueric systems. A typical digital interval timer is shown in block diagram form in Fig 22-1. As indicated in the figure, such a system may include a power supply, a time base oscillator, a multistage counter, a setting mechanism, a decoder to determine when the set time has elapsed, and an amplifier stage which feeds the output transducer (Ref 5)

Foremost in the manufacture and production of various units is the choice of material. It must have sufficient strength to withstand structural and hydraulic forces. Surface hardness, ease of fabrication, working temperature, and type of working fluid are also important (Ref 7)

The use of flueric devices for timing applications in ordnance has the following advantages: 1) High reliability, 2) Large temperature range, 3) Unaffected by electromagnetic and nuclear radiation, 4) Effects of shock and vibration can be reduced to zero by initial choice of materials, 5) Number of system interfaces can be reduced, 6) Potential low cost by technological development, 7) Unaffected by corrosive liquids, 8) Miniaturization. The disadvantages are: 1) Limit of response time on switching speed, 2) Limit of signal propagation, 3) Large power con-

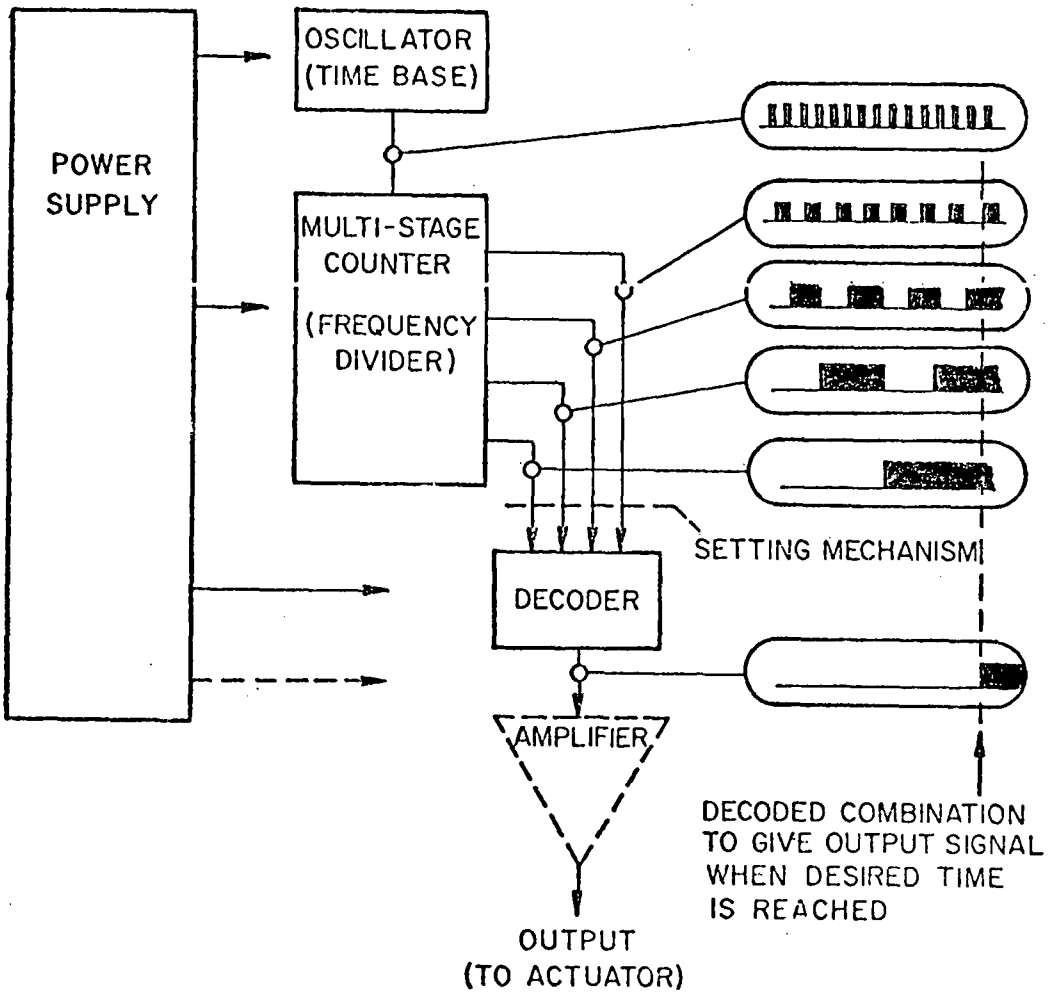


FIG 22–1 Digital interval Timer Block Diagram (Ref 5)

sumption, 4) Need for contaminant-free supply, 5) Dependence on temperature and pressure

In addition, before end items suitable for military application can be developed, the following design limitations must be overcome: 1) Oscillator stability, timer accuracy and reliability under such end item conditions of environment as military temperature range and spin 30000rpm (max for artillery fuzing), 2) Ruggedness, 3) Need for adequate power supply (for up to 200 secs operation), 4) Sample timer setting means increments as small as 0.1 secs

In a typical electronic fuze timer the fundamental components are an oscillator and a binary counter. Using flueric techniques, the oscillator consists of a proportional fluid amplifier with modified sonic feedback loops coupled to a digital fluid amplifier. Fig 8-1 (Ref 7) is a diagram of the amplifiers. The digital amplifier depends upon entrainment in which a stream of fluid flowing close to a surface tends to deflect towards that surface and under proper conditions touches and attaches to the surface. This attachment of the stream to the surface is known as the *Coanda Effect*. Fig 8-1 (A) shows a digital amplifier and Fig 8-1 (B) a proportional amplifier. A fluid oscillator made of both of these components is shown in Fig 8-2 (Ref 7). The binary counters or frequency divider of the timer

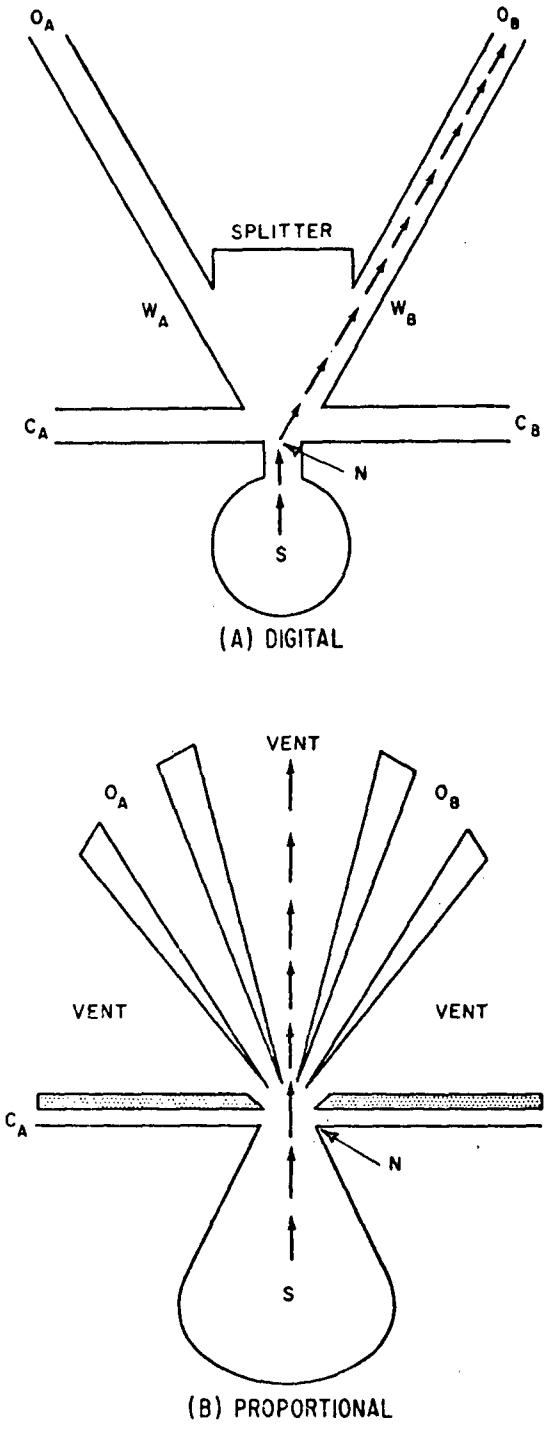


FIG 8–1 Schematic of Flueric Amplifiers (Ref 7)

can be built up of a number of flip-flop stages See Fig 8-3 (Ref 7). These are the basic building blocks for an arming system. Fig 8-4 (Ref 7) shows a timer constructed of units of this type. Fig 24-49 (Ref 4) is a flueric timer packaged for artillery fuze and Fig D a fuze armed by ram air pressure (Addnl Ref D)

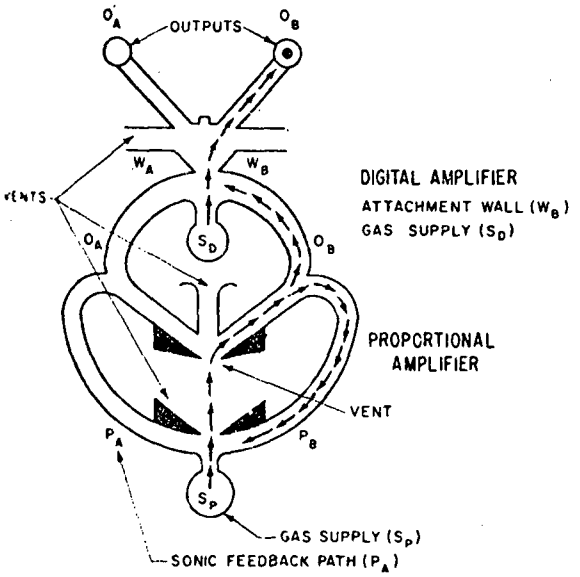


FIG 8–2 Schematic of Flueric Pressure-compensated Oscillator (Ref 7)

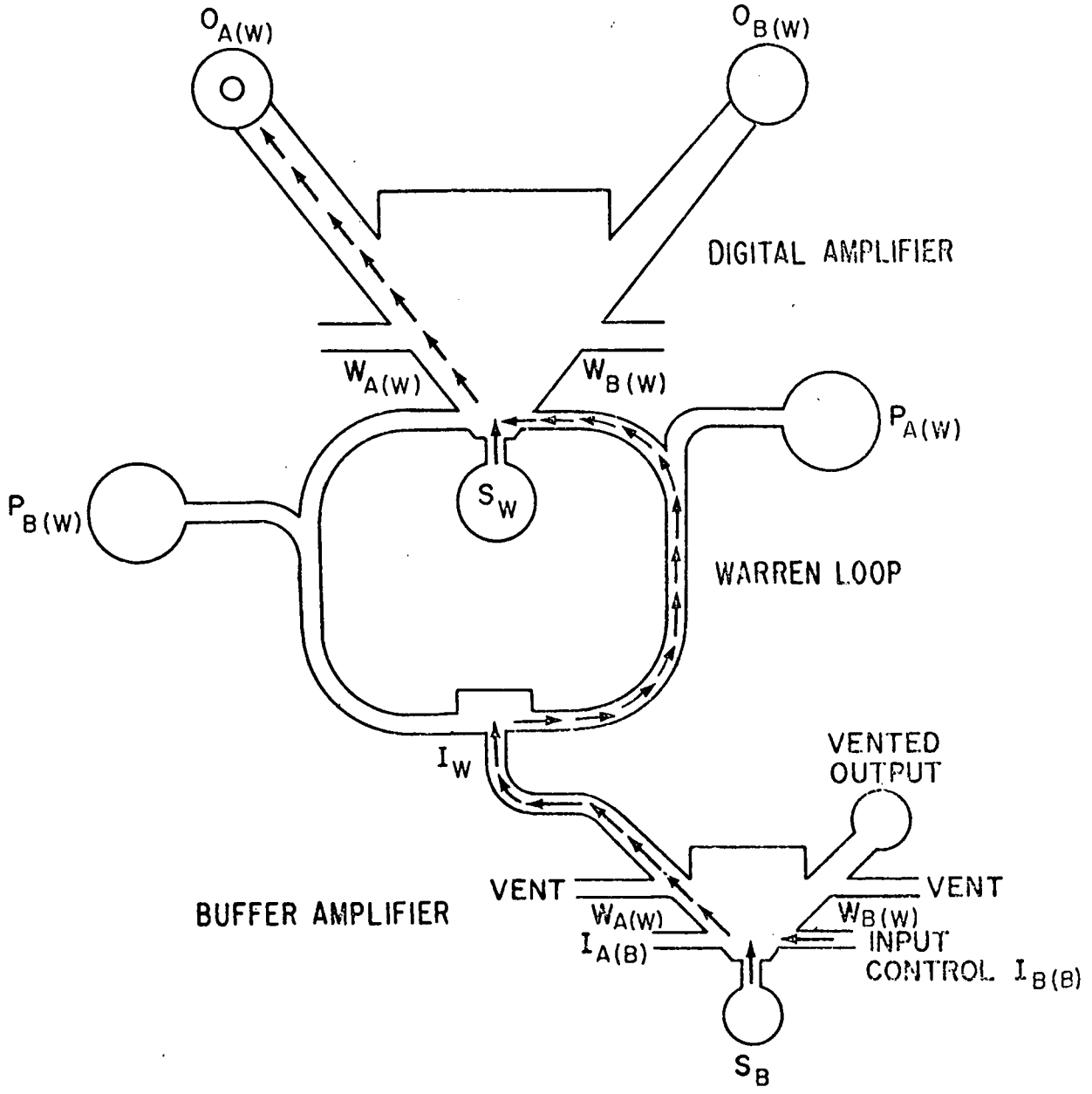


FIG 8–3 Schematic of Flueric Counter Stage
(Ref 7)

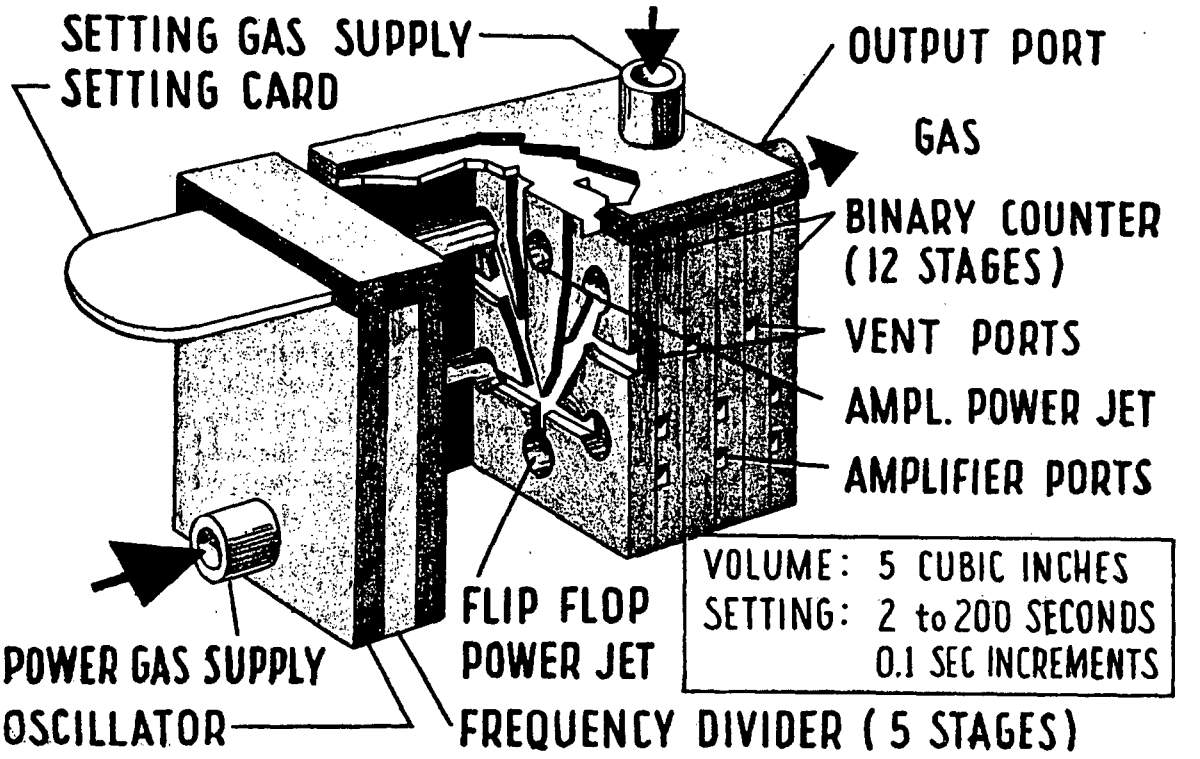


FIG 8–4 Flueric Timer

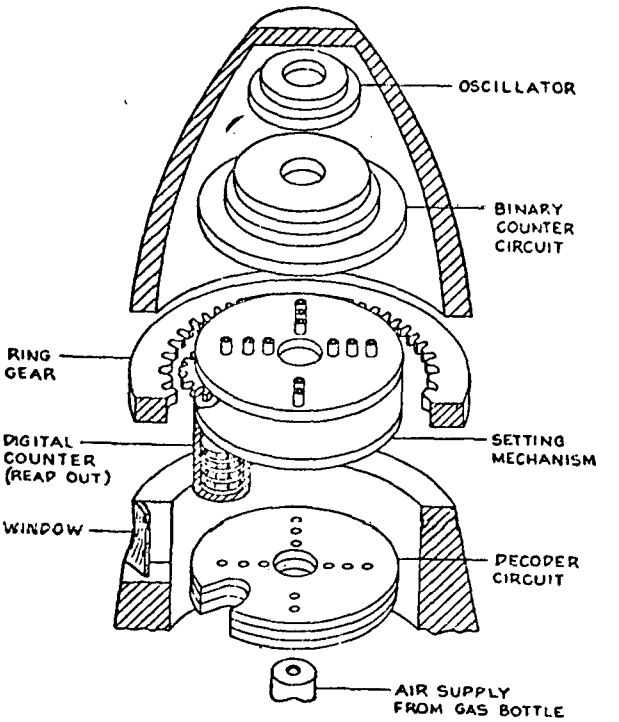
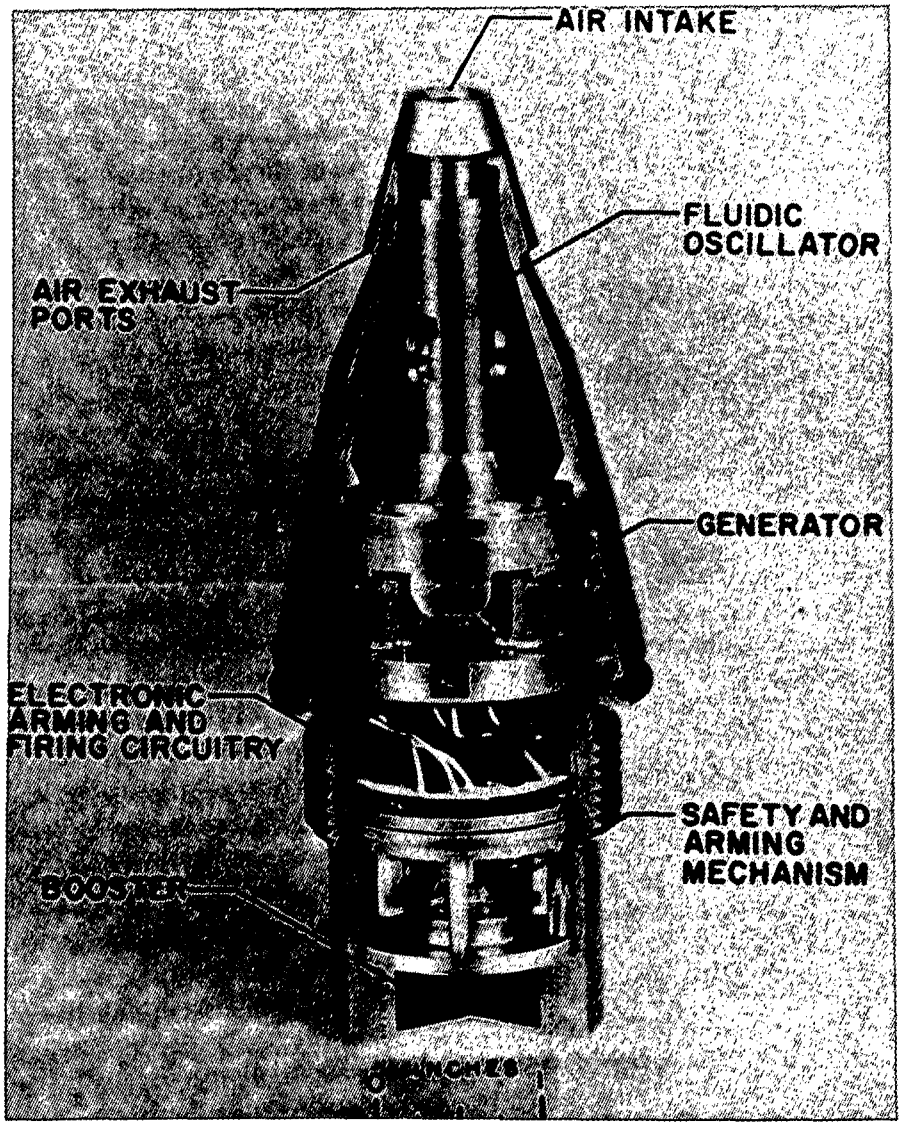


FIG 24–49 Flueric Timer Packaged for Artillery Fuze (Ref 4)



This fuze uses a fluidic power supply driven by ram air pressure; power enough to activate the arming circuit is reached only after a safe distance is traveled.

FIG D
(Addnl Ref D)

Refs: 1) R.L. Daugherty & A.C. Ingersoll, "Fluid Mechanics", McGraw-Hill, NY (1954) 2) H.W. King & E.F. Brater, "Handbook of Hydraulics", 5th edit, McGraw-H ll, NY (1963) 3) R.A. Shaffer, "The Application of Pure Fluids Technology to Artillery Fuzes", FrankfordArsnRept **M66-20**, Philadelphia, Pa (May 1965) (conf) (No classified information taken from this ref) 4) R.A. Shaffer, "Fluid-Mechanical Problems Associated with a Flueric Timer Designed for Artillery Fuze Application", Proc Timers for Ordn Symp, Vol 1, HDL, Washington, DC (Nov 1966), pp 58-78 5) Anon, "The Application of Flueric Devices to Ordnance Timers", JournalArtil **51**, JANAF Fuze Committee (3 May 1967) **AD 834083** 6) C.J. Campagnuolo & S.E. Gehman, "Flueric Pressure and Temperature-Insensitive Oscillator for Timer Application", HDL, Rept **TR 1381**, Washington, DC (Feb 1968) 7) Anon, "Ammunition Series Fuzes", **AMCP 706-210** (1969)
Addnl Refs: A) Anon, "Fluidic Systems

Design Guide", Fluidonics Div of Imperial Eastman Corp, Chicago, Ill (1966), p 99 B) Military Standard, **MIL-STD-1306**, "Fluerics: Terminology and Symbols", Dept of Defense (17 July 1968) C) R.K. Smith, "Fluidics in Ordnance", Ordn **56**, 391–94 (March-April 1972) D) C.H. Staley & A.E. Schmidlin, "Military Fluidics", Ordn **57**, 240–42 (Nov-Dec 1972) E) Anon, "Engineering Design Handbook, Timing Systems and Components", **AMCP 706–205** (Soon to be published) F) Anon, "Engineering Design Handbook, Design Guide for Producibility", **AMCP 706–100** (Soon to be published)

Fluidize. In general to convert to a liquid state; but in recent technology the term refers to processes in which a finely divided solid is caused to behave like a fluid by bringing it into suspension in a moving gas or liquid. The solids so treated are frequently catalysts and hence the term "fluid catalysts". In such a case the fluidized catalyst is brought into intimate contact and causes a desired reaction in the suspending liquid or gas mixture. Local overheating of the catalyst is greatly reduced, and portions of catalyst can be easily removed for regeneration without shutting down the unit. There are also non-catalytic applications in which the fluidized solid enters into direct reaction with the liquid or solid

Ref: CondChemDict (1961), 504-L; 8th edit (1971), 394-R

Fluid Mechanics or Dynamics (Flow of Liquids and Gases) and **Hydraulics.**

Fluid Mechanics is, according to the definition given by Kirk and Othmer, the scientific treatment of the action of forces on fluids, with particular emphasis on fluids in motion

To an engineer working in a chemical or explosives (or ammunition) plant, fluid mechanics is useful not only in predicting friction losses and interconversions of pressure and velocity, but also in producing analogies among the transport of momentum, heat and mass that provide a rational basis for the design of apparatus used in nitration, mixing, purification, distillation, separation, catalytic reactions etc

Hydraulics is that branch of fluid mechanics which treats primarily the water in motion, the works and machinery for conducting or raising it, its use in driving machinery etc

Refs: 1) H. Rouse, "Fluid Mechanics for Hydraulic Engineers", McGraw-Hill, NY (1938) 2) C.N. Cox & F.J. Germano, "Fluid Mechanics", Van Nostrand, NY (1941) 3) B.A. Bakhmeteff, "Mechanics of Turbulent Flow", Princeton Univ Press, Princeton, NJ (1941) 4) R.C. Binder, "Fluid Mechanics", Prentice-Hall, NY (1943) 5) H. Lamb, "Hydrodynamics", Dover Press, NY (1945) 6) H. Rouse, "Elementary Mechanics of Fluids", J. Wiley, NY (1946) 7) W.W. Kraft, IEC **38**, 6–7 & 34 (1946) (Fluid Dynamics with 24 refs) and succeeding years in January numbers under Unit Operations (D.F. Boucher was the author in 1948, 1949; Max Leva in 1950; Max Leva & M. Weintraub in 1951 and 1952), etc 8) J.K. Vennard, "Elementary Fluid Mechanics", J. Wiley, NY (1947) 9) J.C. Hunsaker & B.G. Richtmire, "Engineering Applications of Fluid Mechanics", 10) V.L. Streeter, "Fluid Mechanics", McGraw-Hill, NY (1948) 11) L.K. Spink, "Principles and Practice of Flow Meter Engineering", The Foxboro Co, Foxboro, Mass (1949) 12) H.A. Addison, "Hydraulic Measurements", J. Wiley, NY (1949) 13) J.H. Perry, "Chemical Engineers' Handbook", McGraw-Hill, NY (1950), pp 359–454; T.B. Drew, H.H. Dunkle & R.P. Genereaux, "Flow of Fluids (includes a section on fluid statics, pressure gages and manometers, fluids in motion, flow measurements and pipe & fittings, such as flanges, valves, gaskets etc) 14) H. Rouse, Edit, "Engineering Hydraulics", J. Wiley, NY (1950) 15) Collective, "Some Aspects of Fluid Flow" (Paper presented at a conference organized by the Institute of Physics, Oct 1950), E. Arnold, London (1951) 16) R.E. Kirk & D.F. Othmer, Edits, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **6** (1951), pp 614–648; M. Sauders, Jr, "Fluid Mechanics" (Flow Measurements)

(12 refs) 17) O.W. Eshbach, "Handbook of Engineering Fundamentals", J. Wiley, NY (1952), pp 6-01 to 6-65; V.L. Streeter, "Mechanics of Incompressible Fluids" 18) C.H. McClain, "Fluid Flow in Pipes", Industrial Press, NY (1952) 19) "2nd Midwestern Conference on Fluid Mechanics", Ohio State Univ, Columbus, Ohio (1952) 20) H. Rouse & J.W. Howe, "Basic Mechanics of Fluids", J. Wiley, NY (1953) 21) J.M. Kay, "Fluid Mechanics", Cambridge Univ Press (1957) 22) Perry, 4th edit (1963), pp 5-5 to 5-58 23) Kirk & Othmer, 2nd edit **9**(1966), pp 445–83; M. Saunders, "Fluid Mechanics" (Principles & Flow Measurement)

Fluid Transportation. In chemical industries, the fluids which are transported, range, according to Kirk & Othmer (Ref 7), from gases thru liquids of all viscosities to slurries and sludges

Devices which are used for transporting fluids (liquids and gases) may be divided into pumps, ejectors, injectors, elevators, conveyors, air and gas pressure devices (such as acid eggs, air lifts, pulsometers etc). Pumps may be divided into piston (reciprocating), centrifugal, propeller, rotary-displacement, density, impact and momentum and turbine pumps. Pumps which are used for compressing gases are called "compressors"

Refs: 1) F.A. Kristal & F.A. Annett, "Pumps", McGraw-Hill, NY (1940) 2) J. Reilly & W.N. Rae, "Physico-chemical Methods", VanNostrand, NY (1943), Vol **1**, pp 237–270 3) A.H. Church, "Centrifugal Pumps and Blowers", J. Wiley, NY (1944) 4) P. Sporn, E.R. Ambrose & T. Baumeister, "Heat Pumps", J. Wiley, NY (1947) 5) R.T. Kent, "Mechanical Engineers' Handbook", J. Wiley, NY (1950) 6) J.H. Perry, Edit, "Chemical Engineers' Handbook", McGraw-Hill, NY (1950), pp 1414–39; F.L. Lucker & A.W. Loomis, "Pumping of Liquids" 7) R.E. Kirk & D.F. Othmer, Edits, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **6**(1951), pp 648–55: B.G. Case & M. Souders, Jr, "Fluid Mechanics" (Transportation) 8) L.S. Marks, "Mechanical Engineers' Handbook", McGraw-Hill, NY (1951) 9) Perry, 4th edit (1963), pp 6-2 to 6-15 (Pumping of Liquids) 10) Kirk & Othmer, 2nd edit **9**(1966), pp 473–83; M. Sounders, "Fluid Mechanics" (Flow Measurement)

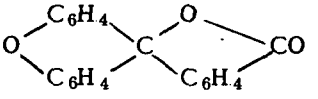
Fluo- or Fluoro-. A prefix indicating the presence of fluorine

Fluoborates or Fluoroborates. See under FLUORINE DERIVATIVES

Fluoboric (or Fluoroboric) Acid. See under FLUORINE DERIVATIVES

Fluoran and Derivatives

Fluoran or *O_2-Phenol-phthalein-anhydride*,



mw 300.30, mp 173–175°, 180–184°, ndls (from alc); sol in nitric & sulfuric acids. Prepd by Baeyer as a by-product of the synthesis of phenolphthalein from phthalic anhydride, phenol, and sulfuric acid, but not purified sufficiently for analysis and identification until six years later. Prepd in about the same way (addn of boric acid) in 8% yield by Copisarow. Prepd by Ferrario from Grignard addn of o-CH_3O-phenyl MgI to phthalic anhydride and acidolysis of the resulting 3,3-bis-(2-methoxy-phenyo)-phthalid with concd HCl at 130–40° (Refs 1, 2, 3, 4, 5, 7, & 8)

x,x,x,x,x-Pentanitrofluoran,
$C_{20}H_7O_3.5NO_2$; mw 525.30, N 13.33%, mp over 335°, plates (from Nitrobenzene+alc), ndls (from acet+w). Prepd by Meyer (Ref 6) by warming fluoran in mixed nitric-sulfuric acid for one-half hour, drawning in water, filtering and recrytg from NB+alc (Refs 1a & 6) Its expl props are not described

Refs: 1) Beil **19**, 146, (676) & [173] 1a) Beil **19**, 149 (Pentanitro) 2) A. Baeyer,

Ber **9**, 1230(1876) 3) A. Baeyer, Ann **202**, 69(1880) 4) A. Baeyer, Ann **212**, 349 (1882) 5) R. Meyer, Ber **24**, 1417(1891) 6) R. Meyer & L. Friedland, Ber **31**, 1744 (1898) 7) E. Ferrario, Gazz **41**, I, 1–11 (1911) 8) M. Copisarow, JChemSoc **117**, 212–13(1920)

Fluoranthene or **Idryl and Derivatives**

Fluoranthene (Called 1,2-benzo-acenaphthylen or 1,8-o-phenylen-naphthylen in Ger), $C_{16}H_{10}$(Thought to be $C_{15}H_{10}$ in early literature), mw 202.24, colorless ndls, sp gr 1.158 at 20°, n_D 1.739 at 20°, bp 393°, 217°(30mm Hg), mp 109.5–110.5°, sol in benz, chlf, eth, HAc & hot alc. A tetracyclic fused ring aromatic hydrocarbon, this material is found in some coal tars. It forms definite complexes with many polynitrated aromatics (Refs 1 to 6)

Refs: 1) Beil **5**, 685, (340, 344), [609] & {2276} 2) J. vonBraun & E. Anton, Ber **62B**, 145(1929) & CA **23**, 2713(1939) 3) T. Sinomiya, BullChemSocJapan **15**, 259–70(1940) & CA **34**, 7900(1940)(Complexes with TNB, TNT, TeNB, etc) 4) M. Orchin & E.O. Woolfolk, JACS **68**, 1727–9(1946) & CA **40**, 7182(1946)(Complex with Trinitrofluorenone) 5) S.H. Tucker & M. Whalley, ChemRevs **50**, 483–538(1952) & CA **46**, 8079 (1952)(The chemistry of fluoranthene, 186 refs) 6) CondChemDict (1961), 504-R; (1971), 394-R

Mononitrofluoranthenes, $C_{16}H_9NO_2$, mw 247.24, N 5.67%:

1-Nitro-, yel prisms, mp 151–53°(Ref 1)
2-Nitro-, yel ndls, mp 153–53.5°(Ref 2)
3-Nitro-, yel crysts, mp 159–60°(Ref 3)
4-Nitro-, yel crysts, mp 162–63°(Ref 4)
8-Nitro-, yel ndls, mp 158–60°(Ref 5)
9-Nitro-, yel ndls, mp 165–66°(Ref 6)

Refs: 1) N. Campbell & J.F.K. Wilshire, JCS **1954**, 867 & CA **49**, 6901(1955) 2) M.C. Kloetzel et al, JACS **78**, 1165(1956) & CA **50**, 12958(1956) 3) Beil **5**, {2279} 4) R.J. Garascia et al, JOC **17**, 266(1952) & CA **47**, 2151(1953) 5) Same as Ref 2 6) N. Campbell et al, ProcRoySocEdinburgh, Sect A**65**, Pt 3, 223(1959–60) & CA **55**, 24704(1961)

Dinitrofluoranthenes, $C_{16}H_8N_2O_4$, mw 292.24, N 9.59%:

1,2-Dinitro-, crysts (Ref 1)
3,9-Dinitro-, yel-orn crysts, mp 275–76°(Ref 2)
2,x-Dinitro-, yel ndls, mp 345–47°(subl)(Ref 3)
3,x-Dinitro-, red ndls, mp 222°(subl)(Ref 4)
4,x-Dinitro-, red-brn crysts, mp 188–89°(Ref 5)

Refs: 1) C. Finger, Ber **104**(8), 2567(1971) & CA **75**, 110094(1971) 2) E.H. Charlesworth & C.U. Lithown, CanJChem **47**(9), 1595(1969) & CA **70**, 114875(1969) 3) Same as Ref 2 4) N.P. Buu-Hoi et al, JCS **1958**, 4309 & CA **53**, 16131(1959) 5) M.I. Shenbor & N.A. Polezhaev, KhimTekhnol **12**, 111–15 (1969) & CA **72**, 21534(1970)

Trinitrofluoranthene, $C_{16}H_7N_3O_6$; mw 337.24, N 12.46%. The earliest ref (2) to this compd describes it as being yel ndls with a mp >300°. In Ref 3 presumably a different isomer is reported as having a mp of 300°. Both were made by nitrating the parent compd

Refs: 1) Beil **5**, 686 2) R. Fittig & F. Gebhard, Ann **193**, 147(1878) 3) M.I. Shenbor & N.A. Polezhaev, KhimTekhnol **12**, 111–15(1969) & CA **72**, 21534(1970)

Tetranitrofluoranthene, $C_{16}H_6N_4O_8$; mw 382.24, N 14.66%. In Ref 2 one isomer is reported as having mp >300°, and another as mp 188–90°; sol in acet. Both were prepd by nitrating the parent compd at RT. Their expl props are not described in CA

Refs: 1) Beil, not found 2) M.I. Shenbor & N.A. Polezhaev, KhimTekhnol **12**, 111–15 (1969) & CA **72**, 21534(1970)

Pentanitrofluoranthene, $C_{16}H_5N_5O_{10}$; mw 427.24, N 16.39%; crysts, mp 299–302°. Prepd by nitrating the parent compd at RT. Its expl props are not described in CA

Refs: 1) Beil, not found 2) M.I. Shenbor & N.A. Polezhaev, KhimTekhnol **12**, 111–15 (1969) & CA **72**, 21634(1970)

"Fluorel" Brand 2141 Elastometer of Minnesota Mining and Mfg Co, St Paul, Minn 55119 is a fully saturated fluorinated polymer contg more than 60% F by wt and is non-flammable. Lt colored gum, sp gr 1.85, shore "A" hard-

ness 40, embitterment temp −50°F, stable in storage and very resistant to strong bases, fuming nitric, fuming sulfuric, acetic and hydrochloric acids; sol in esters and ketones. Outstanding performance in hydraulic fluids and synthetic lubricants at elevated temperatures. Rated for continuous service at +400°, but can withstand +600°F at limited service. Used in gaskets, hoses, wire & fabric coatings, diaphragms, fuel cells, sealants, etc

Ref: CondChemDict (1961), 504-R; (1971), 395-L

Fluorene and Derivatives

Fluorene or α-Diphenylenemethane, $C_6H_4.CH_2.C_6H_4$; mw 166.21, wh plates(alc), sp gr 1.203 at 0/4°, n_D 1.647 at 20°, bp 293–95°, mp 116°, diff sol in cold alc; sol in hot alc, benz, eth, CS_2 & cold chlf; weakly fluorescent when even sl impure. First found (Ref 2) by distillation of hard coal. First prepd (Ref 3) by reacting CH_2Cl_2 with biphenyl and $AlCl_3$. Heat of combustion at const vol is 1585kcal/m. Forms colored complexes with many polynitro aromatics, among which are: 1:1 with picryl chloride, orn-yel ndls, mp 69–70°; 2:3 with TNB, orn-yel plates, mp 105°; 1:1 with TNT, mp 85°; 1:2 with TeNB, orn-red, mp 130°; 1:1 with 2,4,7-trinitrofluorenone, yel-orn, mp 179–79.4°. Because of the difficulty of separating some of the isomers, and of identifying the substitution positions there is considerable confusion in the literature dealing with fluorene derivs. A. Kliegl (Ref 4) states that seven reported cases of isomeric 9- fluorene derivs prior to 1925 are incorrect

Refs: 1) Beil **5**, 625, (300) & [531] 2) M. Berthelot, AnnCh [4] **12**, 222(1867) 3) P. Adam, AnnCh [6] **15**, 253(1888) 4) A. Kliegl, Ber **62B**, 1327–35(1929) & CA **23**, 4690(1929)(Isomerism in 9-fluorene derivatives?) 5) C. Courtot, AnnCh [10] **14**, 5–146(1930) & CA **25**, 508–11(1931)(Fluorene series)(A review) 6) G. Rieveschl & F.E. Ray, ChemRevs **23**, 287–389(1938) (The chemistry of fluorene and its derivatives) 7) T. Sinomiya, BullChemSocJapan **15**, 259–70(1940) & CA **34**, 7900(1940) (Complex with TeNB) 8) M. Orchin & E.O. Woolfolk, JACS **68**, 1727–29(1946) & CA **40**, 7182(1946)(Complex with trinitrofluorenone) 9) CondChemDict(1961), 505-L; (1971), 395-L

4-Azidofluorene, $C_6H_4.CH_2.C_6H_3N_3$; mw 207.24, N 20.29%; crysts, mp 78–81°. Prepd by treating 4-aminofluorene in alc with dil sulfuric acid. Its expl props are not described in CA

Refs: 1) Beil, not found 2) P.A.S. Smith et al, JOC **23**, 526(1958) & CA **52**, 17230 (1958)

9-Azidofluorene, $C_6H_4.CH(N_3).C_6H_4$; mw 207.24, N 20.29%; crysts, mp 43–44°. First prepd by treating 9-bromo-9-trimethylsilylfluorene with sodium azide in hot methanol, more recently by treating 9-bromofluorene itself under the same conditions. Its expl props are not described in CA

Refs: 1) Beil, not found 2) C. Eaborn & R.A. Shaw, JCS **1955**, 1420–25 & CA **50**, 11300(1956) 3) J.P. Anselme, OrgPrep-Proced **1**(3), 201–3(1969) & CA **71**, 70364 (1969)

9-Diazofluorene, $C_6H_4.C{:}N_2.C_6H_4$; mw 192.22, N 14.58%; red ndls, mp 94–5°(dec at 159–60°), sol in alc, benz & eth. Prepd by treating fluorene-9-hydrazone with yel mercuric oxide in benz. Its expl props are not described in CA

Refs: 1) Beil **7**, (252), [408] & {2337} 2) H. Staudinger & O. Kupfer, Ber **44**, 2207 (1911)

9,9-Diazidofluorene, $C_6H_4.C(N_3)_2.C_6H_4$; mw 248.25, N 33.87%. Prepd by treating a 9,9-dihalofluorene with sodium azide in acetonitrile below 0°, but not isolated in the pure state. Its expl props are not described in CA

Refs: 1) Beil, not found 2) L. Barash et al, JACS **89**, 3932–32(1967) & CA **67**, 81676 (1967)

9-Diazo-x,x-dinitrofluorene, $O_2N.C_6H_3.C{:}N_2.C_6H_3.NO_2$; mw 282.22, N 19.86%. Three isomers were prepd from the corresponding 9-hydrazone compds by treatment with silver oxide in refluxing tetrahydrofuran (isomer, mp of hydrazone, mp of diazo, color/form of diazo): *2,4-*, 255–56° dec, 192–92.5° dec, red-orn ndls; *2,5-*, 264–66° dec, 208° dec, orn crysts; *2,7-*, 275–78°dec (crude), 196–99° dec (crude), or crysts. Their expl props are not described in CA

Refs: 1) Beil, not found 2) F.F. Guzik & A.K. Colter, CanJChem **43**(5), 1441–47(1965) & CA **63**, 450(1965)

9-Diazo-2,4,7-trinitrofluorene, $O_2N.C_6H_3.C{:}N_2.C_6H_2(NO_2)_2$; mw 327.22, N 21.41%; orn ndls, mp 204–05° dec. Prepd from the corresponding hydrazone (mp 250–02°) by oxidation with silver oxide. Its expl props are not described in CA

Refs: 1) Beil, not found 2) A.K. Colter & S.S. Wang, JOC **27**, 1517–20(1962) & CA **57**, 2157(1962)

9-Nitratofluorene, $C_6H_4.CH(ONO_2).C_6H_4$; mw 227.22, N 6.17%; crysts, mp 89°. Prepd by treating 9-bromofluorene with silver nitrate in acetonitrile. Its expl props are not described in CA

Refs: 1) Beil, not found 2) C. Eaborn & R.A. Shaw, JCS **1955**, 1420–25 & CA **50**, 11300(1956)

Mononitrofluorenes, $C_{13}H_9NO_2$, mw 211.22, N 6.63%:

1-Nitro-, crysts, mp 104–06°(Ref 1)

2-Nitro-, ndls, mp 156°(Ref 2)

3-Nitro-, yel ndls, mp 105°(Ref 3)

4-Nitro-, yel ndls, mp 72–5°(Ref 4)

9-Nitro-, plts, mp 181–82°(dec)(Ref 5)

Refs: 1) Y. Yost, J MedChem **12**, 961(1969) & CA **71**, 112683(1969) 2) Beil **5**, 685 & [535] & Y. Yost (Same as Ref 1) 3) Same as Ref 1 4) Same as Ref 1 5) C.D. Nenitzescu & D.A. Isacescu, Ber **63**, 2489 (1930) & CA **25**, 925(1931)

Dinitrofluorenes, $C_{13}H_8N_2O_4$, mw 256.22, N 10.93%:

2,5-Dinitro-, yel ndls, mp 209°(Ref 1)

2,7-Dinitro-, col crysts, mp 333–34°(310°) (Ref 2)

2,9-Dinitro-, crysts, mp 136–37°(Ref 3)

9,9-Dinitro-, ndls, mp 130–131.5°(dec)(Ref 4)

Refs: 1) Beil **5**, [536] and C. Courtot & J. Moreaux, CR **217**, 453(1943) & CA **39**, 1638 (1945) 2) Beil **5**, 629 & [536] and C. Courtot & J. Moreaux, CR **217**, 453(1943) & CA **39**, 1638(1945) 3) Beil **5**, [536] and C. Eaborn & R.A. Shaw, JCS **1955**, 1420 & CA **50**, 11300 (1956) 4) Beil **5**, (302) and H. Schechter & R.B. Kaplan, USP 2997504(1061) & CA **58**, 5516(1963)

Trinitrofluorenes, $C_{13}H_7N_3O_6$

Tetranitrofluorenes, $C_{13}H_6N_4O_8$ are not found in the literature

Fluorenone and Derivatives

Fluorenone (Fluorenon, 9-Oxo-fluoren, Diphenylenketon in Ger), $C_6H_4.CO.C_6H_4$; mw 180.21, yel crysts (alc), sp gr 1.130 at 99.4/4°, mp 85–6°, bp 341.5°, n_D 1.6369 at 99.4°, solubility in g/100g at 23°: 7.5 in 95% alc, 19 in CCl_4, 31 in eth, 68 in benz. First prepd by oxidizing fluorene in hot acetic acid with sodium dichromate. Forms a 1:1 complex with nitric acid at 0°, orn-red ndls, mp 63–4°; also forms a 1:1 complex with $HClO_4$. Forms a 2:1 complex with TNB, mp ca 79°

Refs: 1) Beil **7**, 465, (250), [405] & {2330} 2) C. Graebe & A.S. Rateanu, Ann **279**, 258 (1894)

2-Azidofluorenone, $C_6H_4.CO.C_6H_3N_3$; mw 221.22, N 19.00%; yel plates, mp 117°(expl over 117°). Prepd by treating fluorenone-2-diazonium chloride with sodium azide in water

Refs: 1) Beil **7**, [410] 2) A. Korczynski, BullChemSocFr [4] **35**, 1191–92(1924)

4-Azidofluorenone, $C_6H_4.CO.C_6H_3N_3$; mw 221.22, N 19.00%; crysts, mp 118.5–120°. Prepd by treating 4-aminofluorenone with aq alc sulfuric acid. Its expl props are not described in CA

Refs: 1) Beil, not found 2) P.A.S. Smith et al, JOC **23**, 526(1958) & CA **52**, 17230(1958)

2,7-Diazidofluorenone, $N_3C_6H_3.CO.C_6H_3N_3$; mw 262.23, N 32.06%. Mentioned in a patent disclosure application as a possible photographic sensitizer. Its expl props are not described in CA
Refs: 1) Beil, not found 2) H. Kempfer et al, GerOffen 1925998(1970) & CA **75**, 28234(1971)

Mononitrofluorenones, $C_{13}H_7NO_3$, mw 225.21, N 6.22%:
1-Nitro-, yel crysts, mp 191°(Ref 1)
2-Nitro-, yel crysts, mp 222–23°(Ref 2)
3-Nitro-, crysts, mp 239–40°(Ref 3)
4-Nitro-, yel ndls, mp 174–75.5°(Ref 4)
Refs: 1) E.H. Huntress et al, JACS **75**, 743 (1953) & CA **49**, 2385(1955) 2) Beil **7**, 469, (254), [409] & {2344} 3) Beil **7**, 469 & [410] & {2344} 4) Beil **7**, 469 & [410]

Dinitrofluorenones, $C_{13}H_6N_2O_5$, mw 270.21, N 10.37%:
1,8-Dinitro-, crysts (Ref 1)
2,4-Dinitro-, yel ndls, mp 197°(Ref 2)
2,5-Dinitro-, yel ndls, mp 241°(Ref 3)
2,6-Dinitro-, yel ndls, mp 236–37°(Ref 4)
2,7-Dinitro-, yel ndls, mp 292°(Ref 5)
3,6-Dinitro-, ye, lflts, mp 344–46°(Ref 6)
4,5-Dinitro-, orn ndls, mp 273.5°(Ref 7)
x,x-Dinitro-, yel ndls, mp 220°(Ref 8)
Refs: 1) Beil **7**, 470 & [409] 2) Beil **7**, 470 & {2346} 3) Beil **7**, [410] & {2346} 4) N. Ishikawa & M. Hayashi, Yûki Gosei-Kagaku Kyôkai shi **14**, 80(1956) & CA **51**, 8050(1957) 5) Beil **7**, 470, (254), [410] & {2347} 6) A. Barker & C. Barker, JCS **1954**, 870 & CA **49**, 5399(1955) 7) Beil **7**, 470 & {2346 & 2348} 8) Beil **7**, 470 & {2346} and C. Courtot & J. Moreaux, CR **217**, 453(1943) & CA **39**, 1638(1945)

2,3,7-Trinitrofluorenone, $O_2NC_6H_3.CO.C_6H_2(NO_2)_2$; mw 315.21, N 13.33%. First reported in Ref 2, later shown to be the 2,4,7- isomer. Not otherwise found in Beil or CA
Refs: 1) Beil **7**, 471, (254), [410] & {2348} 2) J. Schmidt & K. Bauer, Ber **38**, 3760(1905)

2,4,7-Trinitrofluorenone, $O_2NC_6H_3.CO.C_6H_2(NO_2)_2$; mw 315.21, N 13.33%; yel ndls, mp 180–81°(176°); sol in acet, benz & chlf; sl sol in alc, eth & HAc. Prepd by hot nitration of fluorenone or 2,7-dinitrofluorenone with mixed acid. Originally thought to be the 2,3,7-. Has some use as a complexing agent for derivatizing aromatic compds *Refs:* 1) Beil **7**, 471, (254), [410] & {2348} 2) J. Schmidt & K. Bauer, Ber **38**, 3760(1905)

2,3,6,7-Tetranitrofluorenone. Reported to have been prepd in Ref 2, later (Ref 3) shown to be the 2,4,5,7- isomer. Ref 4 mentions it as a reactant, but gives no prepn or props
Refs: 1) Beil **7**, (254), [410] & {2351}
2) J. Schmidt et al, Ann **390**, 229(1912)
3) F.E. Ray & W.C. Francis, JOC **8**, 52–9 (1943) & CA **37**, 2363(1943) 4) F.G. Badder & S.A.M. Sherif, JCS **1956**, 4292–95 & CA **51**, 4334(1957)

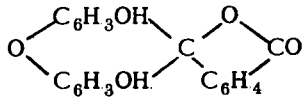
2,4,5,7-Tetranitrofluorenone, $(O_2N)_2C_6H_2.CO.C_6H_2(NO_2)_2$; mw 360.20, N 15.56%; yel ndls(alc), orn plates (HAc) with ½ or 1 HAc of crystn, mp 253°(HAc is lost at 120°); sl sol in HAc. Prepd by hot mixed acid nitration of fluorenone. Originally thought to be the 2,3,6,7- isomer. Later shown to be the 2,4,5,7- isomer by prepn by hot mixed acid nitration of either the 4,5-dinitro- or the 2,4,7-trinitrofluorenones (Ref 3). Has some use as a complexing agent for derivatizing aromatic compds. Its expl props are not described
Refs: 1) Beil **7**, (254), [410] & {2351}
2) J. Schmidt et al, Ann **390**, 229(1912)
3) F.E. Ray & W.C. Francis, JOC **8**, 52–9 (1943) & CA **37**, 2363(1943) 4) M.S. Newman & W.B. Lutz, JACS **78**, 2469–73(1956) & CA **51**, 334(1957)

Fluorenone Peroxide and Related Compounds. In 1940 Wittig and Pieper reported the prepn of *fluorenone (per)oxide*, $C_6H_4.C:O_2.C_6H_4$,

by reacting fluorenone (FL) in ethereal H_2O_2/P_2O_5; it was described as stable yel crysts, mp 108–08.5° (dec over 115°), being sol in methanol, diff sol in benz. In 1949 Criegee et al showed by more exact analysis and various transformations that this compd was actually a *bis(fluorenone complex* of *9,9-bis(hydroperoxy)fluorene*, $C_6H_4.C(O_2H)_2.C_6H_4 \cdot 2FL$, mw 590.64(A). By heating (A) at 85–90° in vacuo, they obtained another complex (B), $(C_6H_4.C[(O_2H)O].C_6H_4)_2 \cdot FL$, mw 606.64; yel crysts, mp 134–35°, explodes in a flame. They converted (B) by treating it with $Pb(Ac)_4$ in HAc to **fluorene-9 9-bisperoxide**, $(C_6H_4.C[(O)O].C_6H_4)_2$; mw 392.42; yel prisms, mp 203° (dec; explodes weakly on confined heating). Treatment of (A) with C_6H_5COCl in pyridine at 0° gave **9,9-bis(benzoylperoxy)-fluorene**, mw 438.44, colorless rhom prisms, mp 106°, explodes over 106°; with p-$O_2NC_6H_4COCl$, **9,9-bis(p-nitrobenzoylperoxy)-fluorene**, mw 528.44; yel crysts, mp 149–50° (sl impure), explodes over 150°, insol in all usual organics. The same treatment of (B) gave **9,9′-benzoylperoxy-9,9′-fluorene peroxide**, mw 634.65, colorless cryst, mp 135° (deton9

Refs: 1) Beil **7**, {2334}2) G. Wittig & G. Pieper, Ber **73**(B), 295–97(1940) & CA **34**, 5834(1940) 3) R. Criegee et al, Ann **565**, 7–21(1949) & CA **44**, 1917(1950) 4) G. Lohaus, Ann **583**, 6–12(1953) & CA **49**, 1588(1955)(Prepn of the bisperoxide by ozonizing 9,9′-bifluorenyl)

Fluorescein *(Fluorandiol, Dihydroxyfluoran, Resorcinol-phthalein or Uranin A)*,



mw 332.30, orn-red pdr, mp dec >290°; insol in cold w; v sl sol in hot w; sol in hot alc & hot AcOH; sol in dil alkalies and acids. Can be prepd by heating phthalic anhydride with resorcinol. Used for dying sea water for spotting purposes, as tracer to locate impurities in wells and as indicator: pH 3.6(yellow) to pH 5.6 (fluorescent). Its very dilute alkaline solns exhibit a very intense greenish-yel fluorescence by reflected light, while the soln is reddish-orn by transmitted light

Forms numerous salts called fluoresceinates

Refs: 1) Beil **19**, 222 2) Hackh's Dict(1944); 547-R 3) CondChemDict (1961), 505-L; 8th edit (1971), 395-L

Fluoresceinates of aluminum, copper and mercury were proposed as fuels in smoke generating compns

Ref: J. DeMent, USP 2995526(1961)

Fluorescence, Luminescence and Phosphorescence.

Fluorescence is the property of certain substances (solids, liquids or gases) to radiate when illuminated, an unpolarized light of a different, usually greater, wavelength. It is due to the return of the electrons displaced by the exciting radiation to a more stable position. The substances having this property are known as *phosphors*, the term usually being restricted to those solids that absorb UV and emit visible light. In usual fluorescent lighting, the tube contains mercury vapor and argon, and the inside of the walls are coated with the fluorescent substance. The passage of an electric current thru the Hg-Ar mixt produces invisible UV light which is absorbed by the phosphor (coating on the wall) and reemitted as visible light. The whole process occurs at a relatively low temperature (hence called a "cold light" process). Besides its use for lighting, fluorescence is used to identify and analyze certain minerals and also some org derivatives

Fluorescence differs from phosphorescence (See below) where the luminescence (See below) continues, even after illumination has ceased

Luminescence is the emission of light at RT under the influence of various physical agents: as mechanical(tribo-l), electrical(electro-l), radiant(photo-l), thermal(thermo-l), or chemical (chemo-l) means. The exciting source also may consist of moving charged particles, such as alpha-, beta-, or gamma-. Certain substances luminesce on crystallization, as,

for example, on crystn of arsenous acid from HCl-soln. Luminescence produced during decaying of wood is attributed to very slow chemical process and luminescence produced by living organisms is attributed to biological process (bioluminescence). If these processes are continuous, they belong to phosphorescence

Phosphorescence is fluorescence that continues for more than a very short time (10^{-6} seconds) after exciting radiation is stopped. As an example, may be cited the faint green glow due to oxidation of white phosphorus on its exposure to air. Also luminosity produced by organisms, such as glow-worms, deep-sea fishes and fireflies. Some mushrooms glow in the dark during their decaying

Refs: 1) Hackh's Dict (1944), 348-L (Fluorescence), 500-R (Luminescence) & 682-R (Phosphorescence) 2) CondChemDict (1961), 505-L (Fluorescence), 682-R)Luminescence) & 888-R (Phosphorescence) 3) Kirk & Othmer, NewEdn, Vol **9**(1966), 485–95 (Fluorescence), **12**(1967), pp 616–25 (Luminescence) & 625–31 (Phosphorescence)

Fluorescence Spectroscopy and Test. *Fluorescence Spectroscopy* is the branch of visible spectroscopy dealing with fluorescence

In conducting a test, the object to be studied (such as an inorg or organic specimen) is shielded from extraneous light and is then illuminated with an ultraviolet lamp (such as a quartz mercury lamp), covered with a filter to remove visible radiation. If the sample glows (fluoresces), the spectrum of this glow is studied by spectroscope and this permits the establishment of the identity of the sample

Ref: G.R. Harrison, R.C. Lord & J.H. Loofbourow, "Practical Spectroscopy", Prentice-Hall, NY(1948), p 14

Fluorescein or Resorcinol Phthalein, $C_{20}H_{14}O_5$; mw 334.32, colorless ndls (from AcOH), mp (for solvent-free) 253–54°; insol in w; sol in alc, eth, AcOH & alkalies. Can be prepd by reduction of fluorescein or by heating p-thalic anhydride & resorcinol. Used as a reagent and in medicine; also as a dye (Refs 1, 2, 3 & 4)

Refs: 1) Beil **18**, 358, (465) & [307] 2) A. Baeyer, ChemBer **4**, 555, 658(1871) 3) CondChemDict (1961), 505-L & R; (1971), 395-L 4) Kirk & Othmer **22**(1970), p 340

Fluorescent Screen. A glass plate covered with a fluorescent substance, such as tungstate or platinocyanide. Used for making visible those rays which are normally invisible to the eye

Ref: Hackh'sDict (1944), 348-R

Fluoric Acid. It could be applied to HFO_3 if it existed, but now it is sometimes erroneously used for hydrofluoric acid, HF

Fluoride. A salt of hydrofluoric acid contg the monovalent F-radical. The term *acid fluoride* refers to a salt of the type MHF_2

Ref: Hackh'sDict (1944), 348-L

Fluorides. See under FLUORINE DERIVATIVES

Fluorinated Ethylene-propylene Resin (FEPR). A copolymer of tetrafluoroethylene, $F_2C{:}CF_2$ and hexafluoropropylene, $F_3C.CF{:}CF_2$, which can be melt processed in conventional molding and extrusion equipment for use as wire insulation, cable jacketing, shaped objects, gaskets, seals, etc. Stands temps up to 200°C and is tough at low temps

Ref: CondChemDict (1961), 505-R; (1971), 395-R

Fluorinated Paraffin. A paraffin oil or wax which has been fluorinated (part of the hydrogen replaced with fluorine) usually by substitution of F in a chlorinated paraffin. Used as inert lubricant and sealant; also as heat transfer medium

Ref: CondChemDict (1961), 505-R

Fluorinated Polymeric Peroxides. 1,1,4,4-Tetrafluorobutadiene is shaken with O under pressure at RT to produce polymeric peroxides contg as units the structures $[CF_2CH:CHCF_2OO]$ and $[CH(CH:CF_2)CF_2OO]$. The amt of O in the copolymers may be varied over wide limits. The polymers are white solids, sol in Me_2CO, which liberate iodine from KI in aqueous Me_2CO and explode at 122°C. They are useful in rocket propellants

Ref: J.L. Anderson & R.C. Putnam, USP 2971949 (1961) & CA **55**, 14918 (1961)

Fluorinating Agent. A compd used to introduce fluorine into some other compd. The higher fluorides of cobalt (CoF_3), manganese (MnO_4), silver (AgF_2) and of chlorine and bromine are used for this purpose. The usual procedure involves replacement of chlorine. Thus, CCl_4 is treated with fluorinating agent to produce CCl_2F_2 or similar compds

See individual fluorine inorganic compds in Ref 2 for their props & uses

Refs: 1) CondChemDict (1961), 505-R 2) Kirk & Othmer, NewEdn, Vol **9** (1966), pp 527ff, "Fluorine Compds, Inorganic"

FLUORINE AND DERIVATIVES

FLUORINE, F_2, mw 38.00; pale-grn, poisonous gas; extremely reactive. Was discovered in 1771 by C.W. Scheele (1742–1786) in fluorite and cryolite and isolated in 1886 by H. Moissan (1852–1907). The density of gas 1.695 (Air 1.0); sp gr of liquid at –188° 1.108; fr p –219.6°; bp –188.1° at 760mm; decompd by w; corrosive and reacts vigorously with most oxidizable substances at RT, frequently with ignition. Forms fluorides with most elements, except the inert gases. Occurs widely distributed to the extent 0.03% of the earth crust. The chief minerals are fluorapatite, cryolite and fluorspar. Can be obtd by electrolysis of molten anhydrous hydrofluoric acid-potassium fluoride melts with special Cu-bearing carbon anodes, steel cathodes, containers and monel screens. Available in special steel cylinders, both as a liquid and as compressed gas

Used as an oxidizer in rocket fuels, production of metallic and other fluorides and fluoro compounds, listed here separately

Refs: 1) Gmelin-Kraut, Syst Number 5 (1959), 1–141 2) R.N. Naszeldine & A.G. Sharpe, "Fluorine and Its Compounds", Mathuen & Co, Ltd, London (1951) 3) C. Slesser & S.P. Schram, "Preparation, Properties and Technology of Fluorine and Organic Fluoro Compounds", McGraw-Hill, NY (1951) 4) M. Stacey, J.C. Tatlow & A.G. Sharpe, Edits, "Advances in Fluorine Chemistry", Vol **1**, Butterworths, London (1960) 5) CondChemDict (1961), 505-R & 506-L; (1971), 395-R 6) R.E. Kirk & D.F. Othmer, Eds, "Encyclopedia of Chemical Technology", Interscience, NY, 2nd edn, Vol **9** (1966), 506–26 7) P. Tarrant, Edit, "Fluorine Chemistry Reviews, Dekker, NY, Vol **6** (1973) 8) H.J. Emeleus & J.C. Tatlow, Editors, "Journal of Fluorine Chemistry", Elsevier, Lausanne, Switzerland (1971)

Addnl Refs:

A) L.R. Brantley, Pacific Rockets **1948** (Summer–Fall), pp 11–16 & CA **44**, 5594 (1950) (Elemental fluorine as oxidizer in rocket engines; various aspects)

B) R.H. Lafferty, Jr et al, C&EN **26**, 3336–37 (1948) & CA **43**, 406 (1949) (Explosibility produced by liquid fluorine brought in contact with organic materials. For example, when neoprene was dipped into liq F at liquid N temp, a sl expln and combstn took place, whereas cotton gave an immediate violent expln with white flame)

C) G.C. Finger et al, JACS **73**, 145–49 (1951) & CA **45**, 7034 (1951) (A compd of formula $C_6HF_2N_3O_3$ is described. It was in the form of orn-red crysts which violently exploded on impact but did not melt below 350°. Its isomer, also explosive, melted at 119.3–120° with decompn)

D) G.A.W. Boehm, Fortune, Dec 1957, pp 170–72 (Fluorine is the best oxidizer after ozone; one of the most corrosive substances known. Specific impulse of F with hydrogen is 374. Less energetic but less cantankerous oxidizers are oxygen difluoride (OF_2) and trioxygen difluoride (O_3F_2)

E) V.I. Siele & H.J. Matsuguma, "Investigation of Synthetic Methods for Preparing the

Fluoro Derivatives of Symmetrical Trinitrobenzene", TechRept **2682**, FREL, Picatinny Arsenal, Project TB3-0115B, Item D, Proj 5, A04-10-006, April 1960
F) E.T. McBee, "Fluorine Containing Explosives Suitable for Explosives and Propellants", Quarterly Progress Report **No 4**, June 1, 1960 to Sept 1, 1960, Purdue Univ, Lafayette, Indiana, Contract No N123(60530) 22662A(Conf) (Not used as a source of information)
G) J.L. Bolvin, "Consideration of Hypothetical Boron and Fluorine Derivatives for High Energy Systems" (U), Armament Research and Development Establishment, Canada, **TM 382/60**, March 1963(Conf)(Not used as a source of info)

FLUORINE DERIVATIVES
(Fluorides and Fluorocompounds)

Their principal parent compound is **Hydrofluoric (or Phthoric) Acid Gas.** (Hydrogen Fluoride), HF, mw 20.01; colorless, very poisonous gas, density 0.713(Air=1.0), sp gr of liquid 0.988 at +13.6°, fr p -83°, bp +19.4°; sol in w or alc. Can be prepd by distilling from a mixture of Ca fluoride (fluorspar) and sulfuric acid. Used as an additive in liquid rocket proplnts and in refining of uranium. Also as fluorinating agent in org and inorg reaction; prepn of Al fluoride, other fluorides, fluoroborates, fluorocarbonates, fluorocarbons and fluorosilicates

Its aqueous soln is colorless, fuming, corrosive acid, which attacks glass and Si-contg materials. Available in commerce in strengths up to 70%. Prepd by absorbing HF-gas in w. Used for polishing, etching and frosting of glass; pickling and electropolishing of metals; purification of filter paper and graphite; dissolution of ores, etc
Refs: 1) Gmelin-Kraut Syst Number 5(1959), 142-220 2) CondChemDict (1961), 586-R (Hydrogen fluoride, anhydrous); (1971), 454-L (Hydrofluoric Acid); 455-R (Hydrogen fluoride, anhydrous)

Aluminum Fluoride Anhydrous, AlF_3, mw 83.93; wh crysts, sp gr 2.882 at 25°; mp-sublimes at about 1260° w/o melting; sl sol in w; sol in acids or alkalies; insol in most org solvents. Prepd by dissolving alumina trihydrate in aq HF, followed by crystn and calcination to remove water. Used in glass industry for manuf of porcelain ware (Refs 1 & 2)

Aluminum Fluoride Hydrate, $AlF_3 . 3.5H_2O$; mw 147.03; wh crystn pdr; mp - loosing $3H_2O$ at 250°; sl sol in w. Prepd by action of aq HF on alumina trihydrate, followed by crystn. Used for prepn of wh enamel
Refs: 1) Gmelin-Kraut Syst Number 35(1934), 156-63 2) CondChemDict (1961), 50-R; (1971), 36-L

Aluminum Fluosilicate or **Aluminum Silicofluoride,** $Al_2(SiF_6)_3$; mw 480.83; wh pdr; sl sol in cold w; sol in hot w. Can be prepd by treating alumina trihydrate with fluosilicic acid. Used for prepn of artificial gems, enamels & glass
Refs: 1) Gmelin-Kraut Syst Number 35(1934), 344 2) CondChemDict (1961), 50-R; (1971), 36-L

Ammonium Fluoride or *Malt Salt,* NH_4F, mw 37.04, wh poisonous crysts; sp gr 1.31, mp - decomp by heat w/o melting; sol in cold w. Prepd by interaction of Amm hydroxide with HF, followed by crystn. Used in glass etching, textile mordant, wood preservation and in analytical chemistry (Refs 1 & 2)

Ammonium Fluosilicate (Cryptohalite), $(NH_4)_2SiF_6$; mw 178.17; wh crystn pdr; sp gr 2.01, mp - sublimes; sol in alc & w. Can be prepd by interaction of Amm hydroxide with fluosilicic acid, followed by crystn. Used for glass etching, casting of light metals and electroplating
Refs: 1) Gmelin-Kraut Syst Number 23(1936), 145 & 414; 2) CondChemDict(1961), 72-R; (1971), 52-L

Azido Fluoride. See Fluorine Azide in Vol 1, p A536-L

Barium Fluoride, BaF_2, mw 175.36; colorless, very poisonous crysts; sp gr 4.826, mp 1280°, bp 2260°; sl sol in w; sol in alc. Can be prepd by the interaction of Ba sulfate and HF, followed by crystn. Used in embalming and manuf of enamels; its single crysts are used for spectroscopy (Refs 1 & 2)

Barium Fluorosilicate or **Barium Silicofluoride,** $BaSiF_6$, mw 279.45, wh crystn pdr. sp gr 4.279 at 15°; nearly insol in w; insol in alc; sl sol in HCl. Can be prepd by treating Ba carbonate with fluosilicic acid. Used as an insecticide
Refs: 1) Gmelin-Kraut Syst Number 30(1960), 318 & 489; 2) CondChemDict(1961), 126-R; (1971), 92-R

Benzotrifluoride or **Trifluoromethylbenzene** (Toluene Trifluoride), $C_6H_5{\cdot}CF_3$, mw 146.11, colorless, flammable liq with aromatic odor, sp gr 1.1812(25°/4), fr p −29.1°, bp 102.1° (760mm), n_D 1.4146 at 20°, fl p 54°F (closed cup); decompd by w; miscible with alc, eth, acetone, benz & CCl_4. Can be prepd by heating benzotrichloride with SbF_3 and by other methods (Ref 1). Used as a solvent, as a dielectric fluid, vulcanizing agent and insecticide
Refs: 1) Beil **5**, 290, (149), [224] & {678} 2) CondChemDict (1961), 141-L; (1971), 103-R

Beryllium (Glucinum) Fluoride, BeF_2, mw 47.01, colorless, poisonous pdr, sp gr 2.01 at 15°, mp 800°, bp – sublimes; v sol in w; sol in alc, acids and alkalies. Can be prepd by thermal decompn (at 900–950°) of ammonium beryllium fluoride. Used in production of beryllium metal by reduction with Mg metal
Refs: 1) Gmelin-Kraut System Number 26(1930), 105 2) Hackh'sDict (1944), 123-R 3) CondChemDict (1971), 109-L

Beryllium-Potassium Fluoride (Potassium-Beryllium Fluoride), $BeF_2{\cdot}2KF$; mw 163.21, colorless, poisonous crysts, sp gr ? , mp ? ; sl sol in w; insol in alc. Can be prepd by mixing aqueous solns of KF & beryllium fluoride (Ref 1)
Refs: 1) Gmelin-Kraut Syst Number 26(1930), 172 2) CondChemDict (1961), 147-L; (1971), 109-R 3) Kirk & Othmer **9**(1966), 553

Beryllium-Sodium Fluoride (Sodium Beryllium Fluoride), $BeF_2{\cdot}2NaF$; mw 131.00; wh or grayish poisonous cryst pdr; mp about 350°, bp – decompd; sl sol in w; insol in alc. Can be prepd by heating an aqueous soln of NaF_2 & beryllium fluoride.(Ref 1). Used for prepn of pure Be metal
Refs: 1) Gmelin-Kraut Syst Number 26(1930), 169 2) CondChemDict (1961), 147-R; (1971), 109-R 3) Kirk & Othmer **9**(1966), 553

Boron Fluoride or **Boron Trifluoride,** BF_3, mw 57.82; colorless gas, density 2.3(Air = 1.0), fr p −126.8°, bp −101°; sol cold w; decomp in hot w; decomp in alc. Can be prepd from B trichloride & anhyd HF or by combination of elements. Used as catalyst in org synthesis and in instruments for measuring neutron intensity

It easily forms double compds, such as boron trifluoride etherate. Another compd, boron trifluoride-monoethylamine, $BF_3{-}C_2H_5NH_2$ is wh to pale tan flakes, melting at 88–90°. It released BF_3 above 110° and is used for elevated temp cure of epoxy resins
Refs: 1) Gmelin-Kraut Syst Number 13(1954), 168 2) CondChemDict (1961), 166-L & R; (1971), 125-L 3) Kirk & Othmer **9**(1966), 554

Boron-Potassium Fluoride. See Fluoborate (Fluorborate) of Potassium

Boron-Sodium Fluoride. See Fluoborate (Fluorborate) of Sodium

Bromine Pentafluoride, BrF_5, mw 174.02, colorless, corrosive liq; sp gr 2.466 at 25°, mp 8.8°, bp 40.5°, vapor press 7psia at 70°F; decomp by w. Can be prepd by reaction of F & KBr in the cold or by heating F & $ZnBr_2$ (Ref 1). Used as oxidizer in liq rocket proplnts
Refs: 1) Gmelin-Kraut Syst Number 7(1931), 338 2) CondChemDict (1961), 171-R 3) Kirk & Othmer **9**(1966), 586

Bromine Trifluoride or **Fluorine Bromide,** BrF_3, mw 136.92, colorless to yel corrosive liq; sp gr 2.803 at 24°, mp 5°, bp 135°, vapor press 15psia at 70°F; decomp violently by w and alkalies. Can be prepd by heating BrF_3 & fluorine at 200°(Ref 1)

Used as fluorinating agent & electrolytic solv
Refs: 1) Gmelin-Kraut Syst Number 7(1931), 337 2) CondChemDict (1961), 171-R; (1971), 128-R 3) Kirk & Othmer **9**(1966), 586

Bromodifluoroethane or **Difluorobromoethane,** $F_2HC.CH_2Br$, mw 144.96; colorless liq, sp gr 1.817 at 20°/4, fr p −74.9°, bp 57.3°; v sl sol in w; miscible with alc & eth. Can be prepd by reaction of difluoroethyl alcohol, phosphorous & bromine
Refs: 1) Beil **1**, 89, (127), [61], {178} & <154> 2) CondChemDict (1961), not listed

Bromotrifluoroethylene (BFE) or **Trifluoromonobromoethylene,** $BrFC:CF_2$; mw 160.94. The name is used both for the monomer and polymers made from it. The polymers are usually clear oils at RT and non cracking solids at −65°F(−54°C). The typical fluorocarbons are chemically inert, thermally stable, and nonflammable. The monomer can be prepd similarly to CFE from tribromotrifluoroethane and zinc. BFE polymers are used as flotation fluids for gyros and accelerometers used in inertial guidance systems. Can also be used as CFE (chlorotrifluoroethylene) polymers, but are more expensive
Refs: 1) Beil **1**, 189 2) CondChemDict (1961), 175-L; (1971), 131-R

Bromotrifluoromethane or **Trifluoromonobromomethane** ("Freon 13B1" or "F−13B1"), $BrCF_3$, mw 148.93; colorless gas, fr p −175°, bp −58°(760mm); nonflammable. Can be prepd by heating trifluoromethane & Br in a pyrex tube at 600°(Ref 1). Used in fire extinguishers and refrigerants
Refs: 1) Beil **1**, {83} & <73> 2) Lange (1961); not listed 3) CondChemDict (1961), 175-L and under "Freon", p 515-L 4) Sax (1968), 1191-R

Calcium Fluoride (Fluorite or Fluorspar), CaF_2, mw 78.08, wh pdr, sp gr 3.18 at 20°, mp 1330°; nearly insol in w; sl sol in alc; reacts with hot concd sulfuric acid to liberate HF. Can be prepd by powdering pure fluorite or by the interaction of soluble Ca salt and NaF. Used in manuf of HF, ceramics, smelting, cements, etching of glass, etc. Clear cryst is used in optical equipment. Was used by E. Turpin in expls named "Fluorine", described in Vol **6**, under "Explosifs de Turpin"
Refs: 1) Gmelin-Kraut Syst Number 28(1956), 1 2) CondChemDict (1961), 201-R & 508 (under Fluorspar); (1971), 155-R

Calcium Fluorosilicate (Calcium Silicofluoride), $CaSiF_6$, mw 182.17; wh crysts, sp gr 2.662 at 17.5° and $CaSiF_6.2H_2O$, mw 218.20, wh crysts, sp gr 2.254; v sl sol in w. Can be prepd by the action of fluorosilicic acid on Ca carbonate, followed by crystn. Used in ceramics
Refs: 1) Gmelin-Kraut Syst Number 28(1961), 1105 2) CondChemDict (1961), 201-R; (1971) 155-R

Carbon Tetrafluoride or **Tetrafluoromethane.** See Vol **2**, p C64-L. It is manufd by the E.I. duPont & Co, under the Trademark "Freon-14" or "F−14". See also CondChemDict (1961), 515-R, under FREON; (1971), 857-R (Tetrafluoromethane)

Carbonyl Fluoride or Carbon Oxyfluoride (Fluoroformyl Fluoride), COF_2, mw 66.01, colorless, very poisonous gas, unstable in presence of w; sp gr 1.139 at −114°; fr p −114°, bp −83°. Can be prepd by action of Ag fluoride on CO. Used in org synthesis; suggested as military poison gas
Refs: 1) Beil **3**, [9] & {22} 2) CondChemDict (1961), 220-R; (1971), 170-R

Chlorine Fluoride. See Chlorine Trifluoride

Chlorine Trifluoride, ClF_3, mw 92.46; nearly colorless or pale grn gas; fr p −83°, bp +11.3°. Extremely reactive, comparable to fluorine; reactions with org compds and with w take place with expl violence. Can be prepd in 99% purity by reaction of chlorine and fluorine at 280° and condensation of the product at −80°. Used as oxidizer in proplnts, in incendiaries and for cutting oil well tubes
Refs: 1) Gmelin-Kraut, not found 2) Lange (1961), 240 3) CondChemDict (1961), 255-R; (1971), 197-R 4) Kirk & Othmer **9** (1966), 586

Chlorobenzotrifluorides, $Cl.C_6H_4.CF_3$, mw 180.56. The following isomers exist: *o-Chlorobenzotrifluoride, o-Chlorotrifluoromethylbenzene* or *o-Chloro-α,α,α-trifluorotoluene*, colorless liq with aromatic odor, sp gr 1.379(15.5°/15.5), fr p −7.4°, bp 152° (760mm), n_D 1.456 at 20°, fl p 59°C(closed cup). Can be prepd by reaction of 2-chloro-

1-trichloromethylbenzene, HF in the presence of PCl_5 & Sb(V) fluoride chloride. Used as intermediate, solvent and dielectric liquid
m-Chlorobenzotrifluoride, m-Chlorotrifluoromethylbenzene or *m-Chloro-a,a,a;trifluorotoluene*, colorless aromatic liq, sp gr 1.351 at 15.5°/15.5, fr p −56°, bp 138°(760mm), n_D 1.446 at 20°, fl p 50°C(closed cup). Can be prepd from Cl & benzotrifluoride in the presence of FeCl (Ref 1). Used in pharmaceuticals, dielectric and insecticides
p-Chlorobenzotrifluoride, p-Chlorotrifluoromethylbenzene or *p-Chloro-a,a,a-trifluorotoluene*, colorless liq w aromatic odor, sp gr 1.353(15.5°/15.5), fr p −36°, bp 139.3°, n_D 1.446 at 20°, fl p 37°C(closed cup). Can be prepd by heating 4-chloro-1-trichloromethylbenzene with SbF_3 (Ref 1). Uses: same as for o-Chlorobenzotrifluoride
Refs: 1) Beil **5**, {692 & 693} 2) CondChemDict (1961), 258-R & 259-L; (1971), 200-R

1-Chloro-1,1-difluoroethane (1,1-Difluoro-1-chloroethane) (Genetron-142-B, Trademark of Esso Standard), $CH_3.CClF_2$; mw 100.50; colorless, flammable gas; sp gr 1.194 at −9°, fr p −130.8°, bp −9.2°; insol in w. Can be prepd by chlorinating 1,1-difluoroethane in UV light. Used as solvent, aerosol proplnt & refrigerant; also for prepn of fluocarbon plastics
Refs: 1) Beil **1**, <138> 2) J.D. Calfee & L.B. Smith, USP 2459767(1949) & CA **43**, 3023(1949) 3) CondChemDict (1961), 260-L & 531-L; (1971), 201-R

1-Chloro-2,2-difluoroethane or **2,2-Difluorochloroethane**, $F_2HC.CH_2Cl$; mw 100.50; colorless gas, sp gr >1, bp 36°. Can be prepd by prolonged heating of 1,1,2-trichloroethane with HF & HgO in an autoclave at 95°(Ref 1)
Refs: 1) Beil **1**, 83, 983 & <138> 2) CondChemDict (1961), not listed

Chlorodifluoromethane or **Difluorochloromethane** ("Freon−22" or Fluorocarbon 22), $CHClF_2$, mw 86.48, colorless, nonflammable gas; sp gr ? , fr p −160°, bp −40.8°. Can be prepd by reaction of chloroform with anhyd HF with Sb chloride catalyst. Used in aerosol propln and refrigerant; also for prepn of tetrafluoroethylene polymers
Refs: 1) Beil **1**, <41> 2) CondChemDict (1961), 260-L & 515-R; (1971), 201-R

1-Chloro-1,2,2-trifluoroethane or **1,2,2-Trifluoro-1-chloroethane**, $F_2HC.CHFCl$, mw 118.49, colorless liq, sp gr 1.365 at 0°, bp 17°; v sol in alc. Can be prepd from acetylene-tetrachloride & $SbCl_5 + SbF_3$ (Ref 1)
Refs: 1) Beil **1**, 83 & <138> 2) Lange (1961), 704

2-Chloro-1,1,2-trifluorethylene (CFE) or **1,1,2-Trifluoro-2-chloroethylene**, $ClFC{:}CF_2$, mw 116.48; colorless, flammagle gas, fr p −157.5°, bp −27.9°; decompd by w. Can be prepd from trichlorotrifluoroethane and zinc. Used for polymerization to colorless oils, greases and waxes; also as refrigerant
Refs: 1) Beil **1**, <646> 2) Lange (1961), 706 3) CondChemDict (1961), 267-R;(1971), 208-R

Chlorotrifluoroethylene Resins. Polymers of CFE characterized by high degree of chemical inertness. Virtually unaffected by inorg acids, alkalies, oxidizing agents, and most org solvents. Typical commercially available materials are: "Fluorothene", "Halon" (Trademark of Allied Chemical Corp, Plastics Div, New York, 10006 and "Kel−F" (Trademark of Minnesota Mining & Mfg Co)
Ref: CondChemDict (1961), 267-R; also 507-R ("Fluorothene"); 559-L ("Halon") and 639-R ("Kel−F"); (1971), 208-R

Chlorotrifluoromethane or **Trifluoromonochloromethane** ("Freon−13"), $CClF_3$, mw 104.47; colorless, non-toxic, nonflammable gas with ethereal odor; fr p −181°, bp −81.4°. Can be prepd from dichlorodifluoromethane in vapor phase with $AlCl_3$ catalyst. Used as a refrigerant- also for hardening of metals and in pharmaceutical processing; and as dielectric & aerospace chemical
Refs: 1) Beil **1**, <42> 2) CondChemDict (1961), 267-R; (1971), 208-R

Chlorotrifluorotrifluoromethylbenzene. See under *Chlorobenzotrifluorides* in this Section

Chlorotrifluorotoluene. See under Chlorobenzotrifluorides in this Section

Chromic Fluoride or **Chromium Trifluoride,** CrF_3, mw 109.01; grn crysts, sp gr 3.78, mp >1000°, bp – decomp; sol in w & acids; insol in alc. Can be prepd by the interaction of Cr hydroxide and HF. Used in moth-proofing woolen fabrics

Forms hydrates $CrF_3.4H_2O$ & $CrF_3.9H_2O$
Ref: CondChemDict (1961), 273-L & R; (1971), 212-L

Cobaltous Fluoride or Cobalt Difluoride, CoF_2, mw 96.94, red-rose crysts or powd, mp ca 1200°, bp 1400°; sol in cold w & hydrofluoric acid; decomp in hot w; highly toxic & irritant. It forms a hydrate, $CoF_2.2H_2O$. Ammine complexes can be prepd from the hydrate
Refs: 1) Sax (1968), 580-L 2) CondChemDict (1971), 223-R

Cobalt Trifluoride, CoF_3, mw 115.94; lt-brn, free-flowing pdr; sp gr 3.88; reacts with w to form bk ppt of cobaltic hydroxide; unstable in moist air. Can be prepd from Co & ClF_3 (Ref 1)
Used as a fluorinating agent
Refs: 1) Gmelin-Kraut Syst Number 58 (1961), 528 2) CondChemDict (1961), 289-L; (1971), 225-R

Copper Fluoride, $CuF_2.2H_2O$; mw 137.57; blue, poisonous crysts; sp gr 2.9; sl sol in w; sol in acids. Can be prepd by treating Cu carbonate with aq HF, followed by crystn. Used in ceramics and enamels
Ref: CondChemDict (1961), 301-R; (1971), 235-L

Copper Fluosilicate or **Copper Silicofluoride,** $CuSiF_6.4H_2O$; mw 277.72; blue, hygroscopic, poisonous crysts; sp gr 2.158, mp – decompd; sol in w, sl sol in alc. Can be prepd by interaction of Cu hydroxide and hydrofluosilicic acid. Used for treating grape vines for "white disease" and for dyeing and hardening white marble (Ref 1)

Lange (Ref 2) also lists $CuFSiF_6.6H_2O$, mw 313.73, blue, delq crysts, sp gr 2.158 at 19°; sol in w
Refs: 1) CondChemDict (1961), 301.R; (1971), 235-L 2) Lange (1961), 248–49

Cyanogen Fluoride or **Fluorine Cyanide,** FCN, mw 45.02; colorless, very irritant gas; forms a wh pulvurelent mass if cooled strongly and sublimes at –72°; insol in w. Can be prepd by interaction of AgF and cyanogen iodide. It attacks glass container under influence of light. Used in org synthesis and as a military poison gas (lachrymator)
Refs: 1) Beil 3, [31] & {75} 2) CondChemDict (1961), 323-R; (1971), 251-L

1,1-Dibromo-2,2-difluoroethane or **2,2-Difluoro-1,2-dibromoethane,** $Br_2HC.CHF_2$; mw 223.87; colorless liq, sp gr 2.312 at 20°, bp 107.2° (760mm); insol in w. Can be prepd by fluorination of 1,1,2,2-tetrabromoethane with anhyd HgF_2 at 150–60° (Ref 1)
Refs: 1) Beil 1, 92, (29), [64] & {189} 2) Lange (1961), 502 3) CondChemDict (1961), not listed

1,2-Dibromo-1,1-difluoroethane or **2,2-difluoro-1,1-dibromoethane,** $F_2BrC.CH_2Br$, mw 223.87; colorless liq, sp gr 2.242 at 12.2°, fr p –56.5°, bp 93° (760mm); insol in w. Can be prepd by reaction of 1,1-difluoroethylene & Br in acetic acid (Ref 1)
Refs: 1) Beil 1, 92, (29), [64], {189} & <160> 2) Lange (1961), 502 3) CondChemDict (1961), not listed

Dibromodifluoromethane, CF_2Br_2, mw 129.93; colorless, poisonous, heavy liq; sp gr 2.288 at 15/4°, fr p –141°, bp +23.2°, n_D 1.399 at 12°; insol in w; sol in methanol & eth. Can be prepd by vapor phase bromination of difluoromethane (Ref 1). Used in prepn of quarternary Amm compds, synthesis of dyes and in pharmaceuticals
Refs: 1) Beil 1, (16), {87} & <80> 2) CondChemDict (1961), 357-L; (1971), 277-R

1,1-Dichloro-2,2-difluoroethane or **2,2-Difluoro-1,1-dichloroethane,** $F_2HC.CHCl_2$; mw 134.95; colorless liq, sp gr 1.494 at 17°, bp 80°; insol in w, miscible with alc & eth. Can be prepd by photochlorination of 1,1-difluoroethane at 200° (Ref 1)

Refs: 1) Beil 1, 85, (24), [55], {151} & <135> 2) Lange (1961), 502

1,2-Dichloro-1,2-difluoroethylene or **1,2-Difluoro-1,2-dichloroethylene,** FClC:CClF, mw 132.93; colorless gas, fr p −112°, bp 20.9°. Can be prepd by gently heating 1,2-difluoro-1,1,2,2-tetrachloroethane with Zn in abs alc to give a mixt of cis & trans forms (Ref 1)
Refs: 1) Beil 1, {658} 2) Lange (1961), 502 3) CondChemDict (1961), not listed

Dichlorodifluoromethane or **"Freon-12".** See in Vol 5 of Encycl, p D1209-L and in CondChemDict (1961), 515-L, under "FREON"; (1971), 285-L

Dichlorofluoromethane or **Fluorodichloromethane** (Freon 21 or "Genetron-21 – Trademark of Esso Standard), $CHCl_2F$; mw 102.93; colorless, nonflammable gas; sp gr 1.426 at 0°; fr p −135°, bp +8.9°; insol in w; sol in alc & in eth. Can be prepd by reaction of chloroform & hydrogen fluoride (Ref 1). Used as solvent, aerosol proplnt, refrigerant, in fire extinguishers and in fluorocarbon plastics
Refs: 1) Beil 1, 61, {47} & <39> 2) CondChemDict (1961), 366-R & 531-L; (1971), 286-L

s-Dichlorotetrafluoroethane or **Tetrafluoro-1,2-dichloroethane** (Fluorocarbon-114 or "Freon-114"), $CClF_2.CClF_2$; mw 170.93; colorless, nearly odorless gas, liq at −94°, bp 3.55°; critical pressure 32.3 atm. Can be prepd by treating perchloroethylene with hydrogen fluoride (Ref 1). See Ref 2 for another method of prepn. Used as aerosol proplnt, solvent, fire extinguisher, refrigerant or solvent
Refs: 1) Beil 1, {152} & <137> 2) DowChemCo, USP 2744148 (1952) 3) CondChemDict (1961), 368-R & 369-L; also 515-R, under "FREON"; (1971), 288-R

Difluoramino Compounds. See Vol 5 of Encycl, p D1258-L. Mentioned in CondChemDict (1961), 805-L as powerful oxiderzers suitable for use in liquid and solid rocket propellants

Difluorenyl-diperoxide. See Vol 5, p D1258-L

Di(fluorenylidenehydro)-sym-tetrazine. See Vol 5, p D1258-L

Difluorobenzene and Derivatives. See Vol 5, pp D1258-R & D1259-R

2,2-Difluoro-1-bromoethane. See 1-Bromo-2,2-difluoroethane in this Section

1,1-Difluoro-1-chloroethane. See 1-Chloro-1,1-difluoroethane in this Section

2,2-Difluoro-1-chloroethane ("Genetron-142B" – Trademark Esso Standard). See 1-Chloro-2,2-difluoroethane in this Section

Difluorochloromethane. See Chlorodifluoromethane in this Section

Difluorodibromoethane. See Dibromodifluoroethane in this Section

Difluorodibromomethane. See Dibromodifluoromethane in this Section

2,2-Difluoro-1,1-dichloroethane. See 1,1-Dichloro-2,2-difluoroethane in this Section

1,2-Difluoro-1,2-dichloroethylene. See 1,2-Dichloro-1,2-difluoroethylene in this Section

Difluorodichloromethane. See Dichlorodifluoromethane in Vol 5 of Encycl, p D1209-L and under "Freon-12" in CondChemDict (1961), 515-L, under "FREON"

Difluorodiphenyltrichloroethane (DFDT) or **1,1,1-Trichloro-2,2-bis(p-fluorophenyl) -ethane,** $(FC_6H_4)_2CHCCl_3$; mw 290.57. Fluorine analog of dichlorodiphenyltrichloroethane (DCDT), misnamed DDT. DFDT first developed in Germany; wh solid with faint odor resembling ripe apples, mp 45.5°; insol in w; sol in acetone, eth, benz, CCl_4, kerosene, dioxane & pyridine. Can be prepd by condensing chloral and fluorbenzene in the presence of sulfuric or chlorosulfonic acid. Used as poison and pesticide
Note: Accdg to USPublicHealthService it is less poisonous to warm-blooded animals and fish than DDT, but does not have a long re-

sidual value. It does not have broad killing power of DDT toward all insects but is more effective against some flying insects, especially house flies
Refs: 1) Beil **5**, {1829} 2) CondChemDict (1961), 346-R; (1971), 269-R 3) Kirk & Othmer, not found

1,1-Difluoroethane ("Genetron-152A" – Trademark of Esso Standard). See Ethylidine Difluoride in this Vol

1,2-Difluoroethane or **Ethylene Fluoride,** $FH_2C.CH_2F$; mw 66.05; colorless gas; bp 31° at 750mm press; d 1.024 at 10°, n_D 1.3014 at 12°; prepd by reacting ethylene bromide & AgF and by other methods (Ref 1)
Refs: 1) Beil **1**, 82, {131} & <121>
2) Lange (1961), 502

1,1-Difluoroethylene, Vinylidene Fluoride or **1,1-Difluoroethane ("Genetron-1132A"** – Trademark of Esso Standard), $H_2C:CF_2$; mw 64.04; colorless gas, fr p –144° at 1 atm, bp –83°; sl sol in w; sol in alc, eth & chlf. Can be prepd by adding HF to acetylene. It is a monomer of growing importance. Used in aerosol proplnts, refrigerants and for prepn of fluorcarbon plastics
Refs: 1) Beil **1**, 186, (77) & {638} 2) Lange (1961), 502 3) CondChemDict (1961), 365-R (Prepn described under Dichlorodifluoromethane); 531-L ("Genetron-1132A"); 1212-L (Vinylidene Fluoride); (1971), 928-R

1,2-Difluoro-1,1,2,2-tetrachloroethane or **1,1,2,2-Tetrachloro-1,2-difluoroethane** ("Freon-BF"), $FCl_2C.CCl_2F$; mw 203.85; colorless crysts, sp gr 1.645 at 20°/4, mp 24.7°, bp 92.8°; insol in w; sol in alc, eth & benz. Can be prepd by heating dichloro-1,2-difluoroethane at 150° (Ref 1). Used as a solvent and as a flash point retarder for hydrocarbons and higher boiling solvents
Refs: 1) Beil **1**, {165} & <146> 2) CondChemDict (1961), 516-L, under "FREON"; (1971), 854-R

Dinitrogen Difluoride or **Difluorodinitrogen,** N_2F_2, mw 66.02; gas which can exist as cis & trans isomers; compd may be toxic. Trans form is used for prepn of ionic fluorine compds; cis form used as polymerization initiator
Refs: 1) Mellor, not found 2) Gmelin-Kraut Syst Number 5 (1959), 246 3) CondChemDict (1961), 805-L (Mentioned as powerful oxidizer suitable for both liquid and solid rocket propellants); (1971), 299-R (Difluorodiazine)

Dinitrogen Tetrafluoride or **Tetrafluorodinitrogen,** N_2F_4, mw 132.04; colorless mobile liq or gas, bp –73°, heat of vaporization 3170 cal/mol; toxic & irritant; explodes in contact with reducing agents at high press. Can be prepd by reaction of nitrogen trifluoride with As at 250–300°, some fluorimide (HNF_2) is produced as by-product. Used as oxidizer in fuels for rockets, missiles and in org synthesis
Refs: 1) Gmelin-Kraut, not found 2) A. Kennedy & C.B. Colburn, JACS **81**, 2906–07 (1959) & CA **53**, 15844 (1959) 3) CondChemDict (1961), 805-L (Mentioned as powerful oxidizer suitable for both liquid and solid rocket propellants); (1971), 857-R (Tetrafluorohydrazine)

Ethylene Fluoride. See 1,2-Difluoroethane

Ethylene Tetrafluoride. See in this Vol, p E291-L

Ethyl Fluoride. See Fluoroethane

Ethylfluoroformate, C_2H_5COOF, mw 92.07; liquid, extremely irritant, sp gr 1.11 at 33°; fr p ?, bp 57°. Can be prepd by interaction of ethylchloroformate and thallium fluoride
Refs: 1) Beil, not found 2) CondChemDict (1961), 470-L; (1971), 368-L

Ferric (or Iron) Fluoride, FeF_3, mw 112.85, grn crysts, sp gr 3.18; sol in w & in acids. Can be prepd by the oxide in aqueous HF. Used in ceramics (porcelain pottery)
Forms the hydrate, $FeF_3.4½H_2O$
Refs: 1) Gmelin-Kraut, Syst Number 59, Teil B (1932), 177 2) CondChemDict (1961), 488-L; (1971), 383-L 3) Kirk & Othmer **9** (1966), 626

Ferrous Fluoride, $FeF_2.8H_2O$, mw 237.98, grn cryst; sol in acids & sl sol in w; its anhy-

drous salt has sp gr 4.09. Can be prepd from the metal or chloride by reaction with HF. Used in ceramics

Refs: 1) Gmelin-Kraut, Syst Number 59, Teil B(1932), 175 2) CondChemDict (1961), 493-R & 494-L; (1971), 386-R 3) Kirk & Othmer **9**(1966), 625

Fluobenzene. See Fluorobenzene

Fluoboric Acid. See Fluoroboric Acid

Fluochemicals. See Fluorochemicals

Fluocompounds. See Fluorocompounds

Fluorimide, HNF_2, mw 53.02; colorless liq, fr p −131°, bp −23°; vapor pressure calcd by Clausius-Clapeyron eqn: logP (mm= −1298/T + 8.072; heat of vaporization (calcd) 5940cal/mole; critical temp 93°

Was obtd during the reaction of nitrogen trifluoride (NF_3) with As at 250–300° to produce dinitrogen tetrafluoride (N_2F_4)

Because of occasional explns, both solid and liquid HNF_2 should be handled with caution

Refs: 1) Gmelin-Kraut Syst Number 5 (1959), 249 2) A. Kennedy & C.B. Colburn, JACS **81**, 2906–07 (1959) & CA **53**, 15844(1959) 3) CondChemDict (1961), not found 4) Kirk & Othmer, not found 5) CondChemDict (1971), not found

Fluorine (Explosif de Turpin). See under Explosifs de Turpin in this Vol, pp E366-L to E367-R

Fluorine Azide or **Azidofluoride.** See Vol 1, p A536-L, under AZIDES, INORGANIC

Fluorine Bromide. See Bromine Trifluoride in this Section

Fluorine Chloride. See Chlorine Trifluoride in this Section

Fluorine Compounds (Danger of Explosion).

Accdg to private communication of Dr J.V.R. Kaufman, formerly of PicArsn and the Minnesota Mining and Manufacturing Co, Minneapolis, Minnesota, caution should be exercised when dealing with some of the fluorine compounds. It was claimed that fully fluorinated lower members, such as $CH_3.CH_2{:}N_2$ are explosively unstable and that treatment of fully fluorinated lower members, such as CF_3COOH with LiAl hydride causes expln. Another fluorine compd which might cause an expln is Ethylene Tetrafluoride, which is described in this Vol, p E291-L

Fluorine Cyanide. See Cyanogen Fluoride

Fluorine Fluorosulfate, SO_3F_2, mw 118.07; decomp at 200° into O & SO_2F_2

Refs: 1) Gmelin-Kraut, not found

Note: Accdg to Dr George H. Cady of the Univ of Washington in Seattle, the compd is probably expl. He said that during the prepn of about 500g of peroxydisulfuryl difluoride (SO_6F_2), about 200g of SO_3F_2 was formed. Sulfuryl fluoride (SO_2F_2) also formed, and also some unidentified compds. The by-products were distilled into a cold, clean, dry steel cylinder strong enough to withstand pressure of 135 atm. (The vapor press of SO_3F_2 at RT is ca 10 atm). When cylinder was brought to RT, the contents of the cylinder exploded. Dr Cady thought that violence of expln suggested a chem expln rather than a simple rupture by gas pressure

Ref: Anon, C&EN, Feb 21 (1966), p 40-R, under BRIEFS

Fluorine Nitrate, Nitrogen Trioxyfluoride or **Nitrosyl Hypofluorite,** NO_3F, mw 81.01; colorless gas, fr p −175°, bp −45.9°, Trouton Constant 20.8. Either liquid, solid or gas, it explodes readily and violently, and often for no apparent reason

Was first obtd in 1935 by Cady (Ref 1a) on treating 3-normal nitric acid with fluorine. Ruff & Kwasnick (Ref 2) showed that it was better to use 100% nitric acid at RT in a quartz apparatus. It can also be made by the action of fluorine on K nitrate

It is a very powerful oxidizer which deflagrates upon contact with reducers such as alcohol, ether & aniline (Ref 7)

Refs: 1) Gmelin-Kraut Syst Number 5 (1959), 254 1a) G.H. Cady, JACS **56**, 2635 (1934) 2) O. Ruff & W. Kwasnick, AngewChem **48**, 238

(1935) 3) D.M. Yost & A. Beerbower, JACS **57**, 782(1935) 4) D.G. Hill & L.A. Bigelow, JACS **59**, 2127(1937) 5) Sidgwick, Chem-Elems **1**(1950), 703 6) CondChemDict (1961), not found 7) R. Schmutzler, AngewChem, IntnlEdit, Engl **7**(6), 440–55(1968)(Review with 228 refs) 7) Sax (1968), 780-L

Fluorine Organic Materials. Experiments were conducted by plunging small pieces of organic materials into 10g of liquid fluorine contained in a small Ni cylinder and surrounded by liquid N in a stainless steel beaker. While neoprene produced only a slight explosion, cotton gave an immediate violent expl with a brilliant white flame

Ref: R.H. Lafferty Jr et al, C&EN **26**, 3336 (1948) & CA **43**, 406(1949)

Fluorine Oxide, F_2O, mw 54.00, colorless gas or yel brn liq, sp gr of liq at –190° 1.65, fr p –224°, bp –145°; v sl sol in w. Can be prepd by hydrolysis of F or by electrolytic methods (Ref 1). It has attracted attention in recent years as an ingredient of high-energy rocket-propellant systems (Ref 3)

Refs: 1) Gmelin-Kraut Syst Number 5(1959), 222 2) Lange (1961), 252 3) Kirk & Othmer **9**(1966), 631 4) CRC Handbook of Chemistry and Physics, The Chemical Rubber Co, Cleveland Ohio, 53rd edn (1972), B-91

Fluorine Ozonide or **Trioxygen Difluoride** (Ozonozonid in Ger), $F–O_3–F$, mw 86.00; solid at bp of nitrogen (77°K) and liquid at bp of oxygen (90°K); decomposes at 120°K. Was prepd by an electric discharge process using a ratio of 3/2 oxygen/fluorine in a tube cooled by liq nitrogen (Ref 6)

It is very reactive substance. A trace amt (0.05% by wt) of F_2O_3 dissolved in liq oxygen makes the resultant oxidizer capable of hypergolic ignition with common hydrocarbons, amine base fuels or hydrogen and provides more stable combstn

Refs: 1) Gmelin-Kraut, not found 2) CondChemDict (1961), not found 3) K.K. Andreev, Explosivst **10**, 47(1962) 4) W.A. Riehl, ARS Journal **32**, 384–88(1962) 5) W.A. Riehl et al, Explosivst **11**, 88(1963) 6) Kirk & Othmer **9**(1966), 633

Fluorine Perchlorate or **Chlorine Tetroxyfluoride,** $FClO_4$, mw 118.46, colorless gas having a pungent acrid odor, fr p –167.3°, bp –15.9°; explodes on slightest contact with rough surfaces, dirt & grease. It is a very dangerous & powerful oxidizer which emits toxic fumes on heating. It was first prepd by reacting gaseous fluorine with $HClO_4$ (Ref 1)

Refs: 1) G.H. Rohrback & G.H. Cady, JACS **69**, 677–78(1957) & CA **41**, 3389(1947) 2) R.L. Farrar Jr, US AtEnergyComm **K-1416**, 1–33(1960) & CA **55**, 6795(1961)(Review on prepn & props) 3) H. Agahigian et al, CanJChem **40**, 157(1962) & CA **56**, 8196(1962) (IR & NMR spectrum) 4) V. Grakauskas, FrP 1360968(1964) & CA **62**, P4949(1965) (Prepn by $F_2 + HClO_4$) 5) Sax(1968), 780-L 6) Y. Macheteau & J. Gillardeau, BullSocChim Fr **1969**(6), 1819–21 & CA **71**, 56207(1969) (Thermal stability at 65–100°)

Fluorine Peroxide or **Dioxygen Fluoride,** $F–O_2–F$, mw 70.00, brn gas, red liq or orn solid; d of liq 1.45 at –57°, d solid 1.912 at –165°; fr p –163.5°, bp –57° best prepd by passing a 1/1 mixt of gaseous O_2 & F_2 at low press thru a high voltage electric discharge tube cooled by liq nitrogen (Ref 2). It is an extremely powerful oxidizing–fluorinating agent and reacts explosively with oxidizing materials

Refs: 1) Gmelin-Kraut Syst Number 5(1959), 232 2) Kirk & Othmer **9**(1966), 633 3) CRC Handbook, 53rd edn (1972), B-91

Fluorines of Turpin. See under Explosifs de Turpin in this Vol, pp E366-L to E367-R

Fluorite. See Fluorspar in this Section

Fluoroacetylene, FC:CH, mw 44.03, colorless gas or liquid; fr p below –196°, bp below –80°. Was prepd in nearly quant yield by pyrolysis of fluoromaleic anhydride in a SiO_2 tube at 650°C and 5–7mm Hg. Liquid fluoroacetylene is treacherously explosive but the vapor is stable (Refs 1 & 2)

It slowly condenses to the trimer 1,2,4-trifluorobenzene, $C_6H_3F_3$

Hg & Ag salts of C_2HF, prepd by passing

its vapor thru aq solns of Hg or Ag nitrates are stable to shock but decomp on warming
Refs: 1) Beil **1**, <957> 2) W.J. Middleton & W.A. Sharkey, JACS **81**, 803–04(1959) & CA **53**, 14028(1959) 3) CondChemDict (1971), not found

Fluoroalcohols. A group of fluorine contg alcohols are available commercially having the general formula $H(CF_2.CF_2)_nCH_2OH$, in which n=1 to 5. They include alcs contg C_3, C_5, C_7, C_9 & C_{11}. Density of liquids 1.48–1.66, n_D 1.318–1.320 (lowest of all known org compds). Derived by free radical telomerization of tetrafluoroethylene with methanol. Used as solvents and in org synthesis
Refs: 1) CondChemDict (1961), 506-L
2) Kirk & Othmer **9**(1966), 751 (Fluoroethanols)

Fluoroaluminate of Potassium or **Potassium Fluoroaluminate** (Ger, Kryolith), K_3AlF_6, mw 258.27; crysts, mp 1025°, heat of formation 44.4kcal/mole; prepd by heating KF & AlF_3 to between 565–840°(Ref 1). Found as a mineral in Germany. Has been used in some propellants and pyrotechnic compns

For example, Brit *Cordite N* contd NC (13.1% N) 19.0, NG 18.5, NGu(Picrite) 54.7, EtCentr 7.5 & K_3AlF_6(called Cryolite in Ref 2) 0.3% with added chalk 0.15%. A similar proplnt known as *Type HPM* contd: NC (13.1% N) 18.7, NG 18.2, NGu 53.8, EtCentr 7.5, K_3AlF_6 0.3 & K sulfate 1.5% with 0.15% chalk added

When incorporated in pyrotechnic compns, it probably imparted pink coloration since Na_3AlF_6 imparted yellow light

The following proplnts were described in a British Report:
Type ASN: NC(12.2% N) 50.00, NG 36.25, EtCentr 5.75, DBuPh 8.7 & K_3AlF_6 4.5% (Do not confuse with ASN described in Vol **1**, p A496-L)
Type HSCK: NC (12.2% N) 49.5, NG 47.0 & EtCentr 3.5, with K_3AlF_6 2.25% added
Refs: 1) Gmelin-Kraut Syst Number 35, Teil B(1934), 450 2) R.A. Connor, Chief of Div 8, "Summary Technical Report of Div 8, NDRC", Vol **1** (1946), 107-L 3) Lange (1961), not found 4) CondChemDict (1961), not found

Fluoroaluminate of Sodium or **Sodium Fluoroaluminate (Cryolite)**, Na_3AlF_6. To the brief description given on p C567-L of Vol **3**, the following uses may be added:
US Cannon Propellants, *M15* and *M17*, described in Specification MIL-P.668A and in Vol **2** of Encycl, p C34
US Cannon Proplnt *M30*, described in Spec MIL-P-46489 and in Vol **2**, p C34
US Cannon proplnts *M31* & *T29*, described on p C34. All of these proplnts contd 0.3% Na_3AlF_6

The following proplnt compns were listed in a British Report
Type N: NC(13.1% N) 19.0, NG 18.7, NGu (Picrite) 55.0 & EtCentr(Carbamite) 7.3% with Na_3AlF_6 0.3% added

Davis (1943), pp 63 & 70–1 lists pyrotechnic compns contg cryolite. Tessier of France introduced it at the time of WWI for the yellow coloring of stars, lances and Bengal lights. Other pyrotechnic uses and several refs are given in Vol **3**, p C567-L

See also Sax (1968), 1097(Sodium Aluminum Fluoride) & CondChemDict (1971), 245-R (Cryolite)

Fluorobenzene and Derivatives
Fluorobenzene or *Phenyl Fluoride*, $F.C_6H_5$; mw 96.10, colorless liq with benz odor, sp gr 1.0252 at 20°/4, fr p –41.9°, bp 84.9°, n_D 1.4677 at 20°; nearly insol in w; miscible with alc & eth. Can be prepd by diazotizing aniline in anhyd HF followed by controlled thermal decompn of the diazonium fluoride (Ref 3). Used as identification reagent for plastic or resin polymers and as insecticide intermediate
Refs: 1) Beil **5**, 198, (108), [147] & {520}
2) CondChemDict (1961), 506-L; (1971), 396-L
3) Kirk & Othmer (1966), 783
(Compare with Difluorobenzene, described in Vol **5**, p D1258-R)

Fluoronitrobenzenes or *Nitrofluorobenzenes*, (ortho-, meta- and para-), $F.C_6H_4(NO_2)$; mw 141.11, N 9.94%. Prepd by variety of nitration methods from fluorobenzene. Its *para*-compd is described in Ref 1, as pale yel

crysts, mp 27°(21.5°), bp 206.7°
Refs: 1) Beil **5**, 241, (128), [180] & {608} (Para deriv) 2) G. Oláh et al, ActaChim-AcadSciHung **7**, 431–42(1955) & CA **53**, 1195 (1959)

Fluoro-2,4-dinitrobenzene or **2,4-Dinitrofluorobenzene,** $F.C_6H_3(NO_2)_2$; mw 186.12, N 15.07%, OB to CO_2 –77.4%; liq, fr p 10–12°, bp 136–38° at 2mm press. Prepd by refluxing 2,4-dichlorobenzene over KF or by nitration with mixed nitric-sulfuric acid and fluorobenzene

It may be a mild expl
Refs: 1) Beil **5**, 262 & (136) 2) Same as Ref 2 of previous item)
(Compare with Dinitrodifluorobenzene in Vol **5**, p D1259-L)

Fluoro-2,4,6-trinitrobenzene or **2,4,6-Trinitrofluorobenzene (TNFB),** $F.C_6H_2(NO_2)_3$; mw 231.13, N 18.20%, OB to CO_2 –45.0%; solid, mp 126°(Ref 6), mp 35°(Ref 4)(?). Prepd by treating 2,4,6-Trinitrochlorobenzene with NaF & HAc (Refs 2 & 3) or by nitration of 2,4-Dinitrofluorobenzene with 60% oleum & sulfuric & fuming nitric acid (Ref 4)

Its prepn was investigated recently at PicArsn (Ref 5) and in Canada (Ref 6). It is an explosive

The method used at PicArsn for prepg TNFB involved the nitration of 2,4-dinitrofluorobenzene, but the attempts, based on literature references, were unsuccessful
Refs: 1) Beil, not found 2) L.G. Wesson, USP 2179605(1940) & CA **34**, 1852(1940) 3) V. Leonard, BritP 537010(1941) & CA **36**, 1496(1942) 4) G. Oláh et al, ActaChim-AcadSciHung **7**, 431–42(1955) & CA **53**, 1195(1959) 5) V.I. Siele & H.J. Matsuguma, "Investigation of Synthetic Methods for Preparing the Fluoroderivatives of Symmetrical Trinitrobenzene"; PicArsnTechRept **2682**, FREL, Project TB 3–0115B, Item D, Project 5A04–10–006, April 1960 6) H.L. Sharma et al, CanadJChem **44**(11), 1327–31 (1966) & CA **65**, 2154(1966)
(Compare with 1,3-Difluoro-2,4,6-trinitrobenzene in Vol **5**, p D1259-L)

Fluorobenzoic Acid and Derivatives
Fluorobenzoic Acid, $F.C_6H_4.CO_2H$; mw 140.11. All three isomers are known: *ortho*, ndls/aq, mp 120–22°; *meta*, lfts/aq, mp 124°; and *para*, mn/aq, mp 184–86°. Methods of prepn are described in Refs:
Refs: 1) Beil **9**, 333, (136, 137), [220] & {1324, 1327} 2) Lange (1961), 554

2-Fluoro-4-nitrobenzoic Acid,
$F.C_6H_3(NO_2).CO_2H$; mw 185.12, N 7.57%, crysts, mp 175°. Prepd by oxidation of 2-fluoro-4-nitrotoluene
Refs: 1) Beil **9**, {1762} 2) H. Goldstein & M. Urvater, Helv **34**, 1350–55(1951) & CA **46**, 5553(1952)

2- Fluoro-4,5-dinitrobenzoic Acid,
$F.C_6H_2(NO_2)_2.CO_2H$; mw 230.13, N 12.19%, OB to CO_2 –62.6%; crysts, mp 137°. Prepd by nitrating with mixed nitric-sulfuric acid in presence of 2-fluoro-4-nitrobenzoic acid
Refs: 1) Beil **9**, not found 2) H. Goldstein & M. Urvater, Helv **34**, 1350–55(1951) & CA **46**, 5553(1952)

Fluoroborate (or Fluoborate) of Potassium. See Potassium Borofluoride in this Section

Fluoroborate (or Fluoborate) of Sodium. See Sodium Borofluoride in this Section

Fluoroboric (or Fluoboric) Acid, HBF_4, mw 87.83; colorless clear, strongly acid liq; bp 130°(dec); miscible with w & alc. Can be prepd by action of boric + sulfuric acid on fluorspar. Used for prepn of fluoborates and stabilized diazo salts. Its specially purified soln used in patented process for electrolytic brightening of Al
Refs: 1) Gmelin-Kraut Syst Number 13(1926), 115 & 13(1954), 189 2) CondChemDict (1961); 504-L 3) Kirk & Othmer **9**(1966), p 563 4) CondChemDict (1971), 394-R

Fluorocarbon Resins. This term includes: polytetrafluoroethylene, polymers of chlorotrifluoroethylene (fluorothene), vinylidene fluoride ($H_2C:CF_2$), hexafluoropropylene (C_3F_6) and similar compds. These polymers are thermoplastic, inert to chemicals and oxidation. They have high heat stability, retain their useful props at both extremely low and high temps, have high electrical resistance to moisture. The materials are available as resins, powders, and dispersions, and as films, sheets, tubes, rods and tapes. Some of them are rubber-like. Commercially available varieties are "Kel-F", "Teflon", "Fluorel", "Aclar" and "Halon"
Ref: CondChemDict (1961), 506-R; (1971), 396-R

Fluorocarbons or Organic Fluorine Compounds. Compds of carbon and fluorine with or w/o hydrogen. They are analogs of hydrocarbons in which all, or nearly all, the hydrogen is replaced by fluorine. They are characterized by extreme chem inertness, do not burn, and are thermally stable to 500°F or more. Can be prepd by various methods all based on the exchange of a halogen atom in an org compd for a fluorine atom in a fluorinating agent. The most common agents are fluorides of alkalie metals (Ref 7). General uses are as aerosol propInts, fire extinguishing agents, lubricants and hydraulic fluids, liquid dielectrics and coolants. There are also some special uses, such as binders in extruded solid propellants
Refs: 1) C. Slesser & S.R. Schram, Eds, "Preparation, Properties and Technology of Fluorine and Organic Fluoro Compounds", McGraw-Hill, NY (1951) 2) W.T. Miller, Jr, NatlNuclearEnergyService, Div VII, **1**, 567–685 (1951) & CA **46**, 7988 (1952) (Numerous fluorocarbons were prepd by polymerization of olefins; some of fluorocarbons proved to be expls sensitive to shock) 3) M.A. Rudner, "Fluorocarbons", Reinhold, NY (1958) (Properties, chemistry, processing, fabrication techniques, and final application of fluorocarbons) 4) CondChemDict (1961), 506-R (Properties and uses of fluorocarbons) 5) G.B. Rice et al, "Extruded Solid Propellants with Fluorocarbon Binders" (U), NavalOrdnanceTestStation, **NOTS TP 3042**, Aug 1963; NAVWEPS Rept **8047** (Conf) (Not used as a source of info) 6) R.E. Banks, "Fluorocarbons and Their Derivatives", Oldboume Press, London, 1965 7) Kirk & Othmer **9**, 686–846 (1966) (Fluorine Compounds, Organic by E.T. McBee et al) 8) L.A. Wall, Ed, "Fluoropolymers", Wiley-Interscience, NY (1971) 9) CondChemDict (1971), 396-L

Fluorocarbon NF Compounds. These compds are discussed in the following Refs
Refs: 1) J.A. Young & R.D. Dresdner, "Fluorocarbon NF Compound", Florida Univ, 3rd QuarterlyTechRept, Jan 1–March 31, 1960, Contract DA–01–009–ORD–772 2) Ibid, 4th QuarterlyTechRept, April 1–June 30, 1960, Contract same as above

Fluorochemicals. Org compds, not necessarily hydrocarbons, in which a large percentage of hydrogen directly attached to carbon has been replaced by fluorine. The presence of 2 or more F atoms on a C atom usually imparts great stability and inertness to the compd and F usually increases the acidity of org acids. The following processes exist for their prepn: a) Electrolysis of solns in HF; b) Replacement of Cl or Br by F with HF in the presence of a catalyst such as antimony trifluoride or pentafluoride; c) Addition of HF to olefins or acetylenes. Used as refrigerants, lubricants, aerosol propInts, fire extinguishers and inert plastics
Refs: 1) CondChemDict (1961), 507-L 2) D.N. Gray et al, "Research on the Synthesis of Fluorochemicals", **APGC TR–59–30**, Proj No 2858, Contract No AF08 (603)–4505, Denver Univ (July 1962) 3) CondChemDict (1971), 396-R

Fluorochemistry and Fluorometric Analysis *Fluorochemistry* is a term proposed in 1942 by J. de Ment for the branch of physicochemical science embracing the theory and applications of luminescence (qv) and radia-

tion (qv) to chemistry. (See also Fluoroscopy, Luminescence etc)

The science is based on the following laws:

1st law of fluorescence (J. deMent, 1942): Before emission can occur from a luminescent system, absorption must first take place

2nd law (Sir G.G. Stokes, 1852): The energy released as luminescence from a body is always less than the energy absorbed for initial excitation

3rd law (J. de Ment, 1943): The absorption of radiation by a luminescent system is a quantum process involving one quantum per absorbing center; the yield of luminescence then, in the ideal case, being unity

Fluorometric Analysis is the branch of analytical chemistry which determines the presence (and the amounts) of certain substances by exposing them to the action of ultraviolet rays (or of X-rays) and observing the fluorescence emitted by them

Refs: 1) M. Haitinger, "Die Fluorescenzanalyse in der Mikrochemie", E. Haim & Co, Wien & Leipzig (1937) 2) C.E. White, IEC (AnalEd) **11**, 63 (1939) (Fluorescent analysis of inorganic materials; includes 26 refs) 3) J.A. Radley & J. Grant, "Fluorescence Analysis in Ultraviolet Light", Chapman & Hall, London (1939) 4) P.W. Danwortt, "Lumineszent-Analyse im filtriertem ultravioletten Licht", AkadVerlag, Leipzig (1940) (Lithoprinted by Edwards Bros, Ann Arbor, Mich) (Contains 1583 refs) 5) J. de Ment, Science **95**, 407 (1942); **96**, 157 (1942) 5a) J. de Ment, JChemEduc **21**, 116–25, 154 (1944) 6) J. de Ment, Mineralogist **11**, 115 (1943) 7) J. de Ment, "Fluorochemistry" (A Comprehensive Study Embracing the Theory and Application of Luminescence and Radiation in Physico-chemical Science), ChemPubCo, Brooklyn (1945) 8) G.R. Fonda, JACS **68**, 347 (1946) 8a) M. Curie, "Fluorescence and Phosphorescence", Hermann, Paris (1946). Reviewed in Nature **160**, 483 (1947) 9) M. Kasha, ChemRevs **41**, 401 (1947) 9a) G.R. Fonda & F. Seitz, "Preparation and Characteristics of Solid Luminescent Materials", J. Wiley, NY (1948) 10) W.E. Forsyth & E.Q. Adams, "Fluorescent and Other Gaseous Discharge Tubes, Murray Hill Books, NY (1948), Chap 8 11) C.E. White, IEC (AnalEd) **21**, 104–108 (1949) (Review of fluorometric analysis; includes 123 refs) 12) R.E. Kirk & D.F. Othmer, Ed, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **4** (1949), p 274. (See also books under Colorimetric Analysis) 13) J.A. Radley & J. Grant, "Fluorescent Analysis in Ultraviolet Light", Van Nostrand, NY (1949) 14) P. Pringsheim, "Fluorescence and Phosphorescence", Interscience, NY (1949) 15) T. Förster, "Fluoreszenz Organischer Verbindungen", Vandenboeck & Ruprecht, Göttingen (1951) 16) E.J. Bowen & F. Wolkes, "Fluorescence of Solutions", Longmans, Green & Co, NY (1953) 17) F. Claisse & C. Samson, "Effects des Heterogeneites en Fluorescence des Rayons X", Ministere des Richesses Naturelles, Quebec (1962) 18) L. Morati, "Elettrofluorescenza Elettroluminescenza", 2nd edit, Hoepli Editore, Milano (1962)

Fluoro Compounds. Compounds contg fluorine show promise as oxidizers. Some fluorine compds presently under investigation include elemental F, halogen fluorides, nitrogen trifluoride, and oxygen difluoride. Fluorides are corrosive and often unstable. Hence, some emphasis is placed on compd stability. In rockets, fluoride fuels offer a good balance between low molecular wt combustion products & high flame temp. Lithium fuels, such as $LiNF_2$, which utilize fluorine, are also of interest

Refs: 1) Anon, "Sources of Energy", US Army Materiel Command Pamphlet **AMCP 706–106** (Aug 1964), p 3-27 (Exotic Propellants) 2) Kirk & Othmer **9** (1966), 527ff

Fluorodichloromethane. See Dichlorofluoromethane in this Section

Fluoroethane or **Ethyl Fluoride,** $H_3C.CH_2F$; mw 48.06; colorless gas, sp gr 1.7 (Air 1.0), fr p −143.2°, bp −37.7°; v sol in w & in alc. Can be prepd by heating bromoethane & HgF at 110° (Ref 1). When heated to decompn it emits toxic fumes of fluorides

Refs: 1) Beil **1**, 82, {130} & <120> 2) Lange (1961), 544(Ethyl Fluoride) 3) Sax (1968), 751-L

Fluoroethylene or **Vinyl Fluoride,** CH_2:CHF; mw 46.04, colored, flammable gas, sp gr 0.853 at −26°, fr p −160.5°, bp −72.2°(−51° in Ref 2); insol in w; sol in alc. Can be prepd by passing a mixt of acetylene & HF over AlF_3 & graphite at 177–400°(Ref 1). The compd is flammable and a dangerous fire & expln hazard. Used as monomer for resins (See Polyvinyl Fluoride)
Refs: 1) Beil **1**, 186, (77) & {637} 2) Cond-ChemDict (1961), 1211-R (Vinyl Fluoride); (1971), 928-L

Fluoroform or **Trifluoromethane** ("Genetron–23" – Trademark of Esso Standard), CHF_3, mw 70.02, gas, sp gr of liq 1.47 at −84°, bp 20° at atm; sol in w & alc; sl sol in chlf. Can be prepd from chloroform HF & NiCl /Al oxide at 280°(Ref 1)
Used as aerosol proplnt, refrigerant, fluoro-carbon plastics etc
Refs: 1) Beil **1**, 59, {34} & <24> 2) Cond ChemDict (1961), 32)-L ("Genetron–23"); (1971, 396-R

Fluoroformyl Fluoride. See Carbonyl Fluoride in this Section

"Fluorolubes". Trademark of Hooker Chem Corp, Niagara Falls, NY, for addition polymers of trifluorovinyl chloride; essentially linear polymers built up of a recurring unit, $-CF_2.CFCl-$. They are odorless, non-toxic, high density, non-flammable, free from hydrogen liquids, stable at temps up to 300°F. They are not attacked by strong acids, alkalies, hydrogen peroxide and other oxidizing agents; insol in petroleum base oils; sl sol in lower alcs; sol in benz, ketones and most chlorinated liquids. Used as lubricant and sealant for plug cocks, valves and vacuum pumps; also gasket and packing impregnant and fluid for hydraulic equipment
Ref: CondChemDict (1961), 507-R; (1971), 397-L

Fluoromethane or **Methyl Fluoride,** CH_3F, mw 34.03; colorless gas, sp gr 0.877, fr p −142°, bp −78.2°; sol in w. Can be prepd from chloromethane & AgF_2 at 225° or from iodo-methane & HgF_2 (Ref 1)
Refs: 1) Beil **1**, 59, (8), [11], {33} & <22>
2) Lange (1961), 604(Methyl Fluoride)
3) CondChemDict (1971), 397-L

Fluorometry is the branch of physics dealing with measuring of X-rays and other radiations by the fluorescence produced
(See also Fluorochemistry, Fluorescence, Luminescence, Radiation and X-rays)

Fluoro-Nitro Compounds. The prepns, structures, physical props & chem props of oxyfluorides of N(NOF) & nitrylhypofluorite (NO_2F); the prepn & props of NOF–HF adducts, the chemistry of NOF.3HF, the NO_2F–HF system, reactions of NOF & NO_2F with acceptor fluorides; addn of NOF & NO_2F across double bonds; N,N-difluorohydroxylamines; and uses of N oxide fluorides are discussed by Schmutzler (Ref 2)
Refs: 1) K. Baum et al, "Research in Fluoro-Nitro Compounds", Aerojet General Corp, Summary Rept **2381**, Sept 1, 1961 thru 31 Aug 1962, Contract Nonr 2655(00)(Conf)(Not used as source of info) 2) R. Schmutzler, Angew-Chem, IntnlEdn, Engl **7**(6), 440–45(1968) & CA **69**, 40751(1968)(Review with 228 refs)

Fluorophenol and Derivatives
Fluorophenol, $F.C_6H_4.OH$, mw 112.10. None described in Ref 3, but *para-* described in Ref 4 as wh crysts, sp gr 1.1889 at 56°, mp 48.2°(stable form), 28.5°(unstable form), bp 185.6°(760mm), 78°(15mm); sol in w. Can be prepd by heating 4-fluoroanisole with aq HI (Ref 2)
Used as intermediate for pharmaceuticals & fungicides and as fungicide
All isomers are described in Refs 1, 1a & 2
Refs: 1) Beil **6**, (97) & {667}(ortho)
1a) Beil **6**, (97), [169] & {668}(meta)
2) Beil **6**, 183, (98), [170] & {669} (para)
3) Lange (1961), not found 4) CondChem-Dict (1961), 507-R (p-Fluorophenol); (1971), 397-L

Fluoronitrophenols or **Nitrofluorophenols,** $F.C_6H_3(NO_2).OH$; mw 157.11, N 8.92%. The following isomers are known:

3-Fluoro-2-nitrophenol, orn-yel ndls, mp 39° (Refs 1 & 6)

4-Fluoro-2-nitrophenol, yel crysts (from alc), mp 73.7°(Ref 2)

5-Fluoro-2-nitrophenol, yel ndls (from petr eth), mp 32°(Ref 3)

6-Fluoro-2-nitrophenol, yel prisms (from di-isopropyl eth), mp 90–91°(Ref 4)

5-Fluoro-3-nitrophenol, yel crysts (from aq HCl or thru sublimation, mp 112°(Ref 4)

2-Fluoro-4-nitrophenol, ndls or tablets (from w), mp 117–18°(Ref 4)

3-Fluoro-4-nitrophenol, ndls (from w or petr eth), mp 42°(Refs 5 & 6)

Other props & methods of prepn are found in Refs

Refs: 1) Beil **6,** [225] & {832} 2) Beil **6,** (121) 3) Beil **6,** [225] & {833} 4) Beil **6,** {833} 5) Beil **6,** [226] & {834} 6) H. Hodgson & J. Nixon, JCS **1928,** 1879–82 & CA **22,** 3643(1928)

Fluorodinitrophenols or **Dinitrofluorophenols,** $F.C_6H_2(NO_2)_2.OH$, mw 202.12, N 13.88%, OB to CO_2 –63.4%. The following isomers are known:

3-Fluoro-2,4-dinitrophenol, yel crysts (from petr eth), mp 138–39°. Prepd by stepwise nitration of m-fluorophenol (Refs 1 & 6)

5-Fluoro-2,4-dinitrophenol, lt-yel microcrysts (from petr eth), mp 138–39°; v sol in w; prepd by nitrating 3-fluorophenol with mixed sulfuric-nitric acid at 10–15° and hydrolizing the reaction product with steam (Refs 1 & 6)

6-Fluoro-2,4-dinitrophenol, yel crysts (from aq alc or benzine), mp 102°; v sol in benz, MeOH & alc; mod sol in w; obtd with other products by nitrating 2-fluoroanisole or 2-fluorophenetol with mixed acid at –5 to 0° (Ref 2)

3-Fluoro-2,6-dinitrophenol, ndls (from petr eth), mp 68.5°; was prepd by stepwise nitration of 3-fluorophenol or 5-fluoro-2-nitrophenol at 10–15°(Refs 3 & 6)

4-Fluoro-2,6-dinitrophenol, yel prisms (from CS_2), mp 50–50.2°; readily sol in eth & alc; sl sol in water; was prepd by nitrating 4-fluorophenetol with concd nitric & sulfuric acids and treating the reaction product with KOH (Ref 4)

3-Fluoro-x,x-dinitrophenol, pltlts (from water), mp 72–74°; was prepd by nitrating 3-fluorophenol with mixed nitric-sulfuric acid (Ref 5). It forms a yel K salt

Refs: 1) Beil **6,** [246] 2) Beil **6,** {869} 3) Beil **6,** [247] 4) Beil **6,** (128) & {870} 5) Beil **6,** 128) 6) H.H. Hodgson & J. Nixon, JChemSoc **1928,** 1879–82 & CA **22,** 3643(1928)

3-Fluoro-2,4,6-trinitrophenol, 3-Fluoropicric Acid or **2,4,6-Trinitrofluorophenol,** $F.C_6H(NO_2)_3.OH$, mw 247.13, N 17.02%, OB to CO_2 –35.6%; crysts (from w), mp 173°. Prepd by stepwise nitration of m-fluorophenol and by other methods (Ref 1). It may be a powerful explosive. It forms an Ag salt, yel ndls; readily sol in aq ammonia & in hot water

Refs: 1) Beil **6,** [283] 2) H.H. Hodgson & J. Nixon, JCS **1928,** 1879–82 & CA **22,** 3643(1928)

Fluoroscopy is the visual examination of X-ray shadow images by means of a fluorescent screen. It may be used for the inspection of light metal castings, some ammunition, castings of explosives etc. In this method, the images are formed directly on a screen and no films are required, which makes the cost of inspection lower than with ordinary X-ray examination

Refs: 1) Hackh's (1944), 349

Fluorosilicates. Salts of Fluosilicic Acid, H_2SiF_6. See Aluminum-, Ammonium-, Barium-, Calcium-, Copper-, Lead-, Magnesium-, Potassium-, Sodium-, and Zinc Fluosilicates in this Section

Fluorosilicic (or Hydrofluosilicic) Acid, (Silicofluoric Acid), H_2SiF_6 ; mw 144.11; colorless, transparent, fuming, corrosive, poisonous liquid; attacks glass & stoneware; kept in wax, wood, or special plastic con-

tainers; sol in w. Obtd as by-product of the action of sulfuric acid on phosphate rock contg fluorides and silica or silicates. Also by action of water on tetrafluoride, SiF_4, which, in turn, is prepd by action of HF on silica. Used for prepn of fluosilicates of Amm, Ba, Cu, Mg, Pb & Zn; also to increase hardness in ceramics, in hardening of cement during building, preserving masonry and as disinfectant

Refs: 1) Gmelin-Kraut Syst Number 15, Teil B (1959), 638 2) CondChemDict (1961), 508-L; (1971), 397-R 3) Kirk & Othmer **9** (1966), p 654ff (Fluosilicic Acid)

Fluorothene. A plastic polymer of trifluorochloroethylene, C_2F_3Cl. Resistant to concd acids and alkalies up to 300°F. Swelled by some chlorinated hydrocarbons

Ref: CondChemDict (1961), 507-R; (1971), 208-R

Fluorotoluene and Derivatives

Fluorotoluene, $F.C_6H_4.CH_3$; mw 110.13. Three isomers o-, m- & p- are known. They are colorless liqs with bp's 113–14°, 115–16° & 116–17°, resp. All isomers are insol in w and sol in alc & eth. Can be prepd from the corresponding toluene-diazonium-tetrafluoroborate

Refs: 1) Beil **5**, 290, (194) & [223] 2) G. Schiemann, ZPhysChem [A] **156**, 397, 417 (1931)

Fluoro-mononitrotoluene, $F.C_6H_3(NO_2).CH_3$; mw 155.13, N 9.03%. The 3-Fluoro-2-nitro-; 4-Fluoro-2-nitro-; 5-Fluoro-2-nitro-; 6-Fluoro-2-nitro-; 4-Fluoro-3-nitro-; 2-, 4- and 6-Fluoro-3-nitro-; and 2- & 3-Fluoro-4-nitrotoluenes are all described in Ref 1. Ref 2 also describes 2-Fluoro-4-nitrotoluene

Refs: 1) Beil **6**, 326, (161), [250, 251] & {742, 743} 2) H. Goldstein & M. Urvater, Helv **34**, 1350–55 (1950) & CA **46**, 5553 (1952) (Its oxidation produced 2-fluoro-4-nitrobenzoic acid)

NOTE: No higher nitrated derivs of Fluorotoluene were found in the literature

Fluorotrichloromethane or **Trichloromonofluoromethane** ("Freon–11" or "Freon–MF", Solvent), CCl_3F, mw 137.38; colorless, volatile liq, sp gr 1.494 at 17.2°, fr p –111°, bp 23.7°, crit press 43.2 atm. Insol in w; miscible with alc & eth. Prepd from CCl_4 & HF in the presence of fluorinating agents, such as antimony tri- and penta-fluorides. Used as aerosol proplnt, refrigerant and fire extinguisher

Refs: 1) Beil **1**, 64, {63} & <54> 2) CondChemDict (1961), 1161-R (Trichlorofluoromethane) and 515-L & 516-L under "FREON"; (1971), 887-L

Fluorspar or **Fluorite** See under Calcium Fluoride in this Section

Fluoryl Perchlorate or **Perchloryl Oxyfluoride.** See Fluorine Perchlorate in this Section

Fluosilicates. See Fluorosilicates in this Section

Fluosilicic Acid. See Fluorosilicic Acid in this Section

"FREON". Trademark of E.I. duPont de Nemours & Co, Inc, Wilmington, Del for a line of fluorinated hydrocarbons used as propellants, refrigerants, blowing agents, fire extinguishers and solvents

The following "Freons" are listed in Ref, pp 515-L to 516-L & other pages

"Freon–11" or "F–11" or *Trichloromonofluoromethane.* See Fluorotrichloromethane in this Section

"Freon–12" or "F–12". See Dichlorodifluoromethane in Vol **5** of Encycl, p D1209-L

"Freon–13", "F–13" or *Monochlorotrifluoromethane.* See Chlorotrifluoromethane in this Section

"Freon–13B1", "F–13B1" or *Monobromotrifluoromethane.* See Bromotrifluoromethane in this Section

"Freon–14" or *"F–14"* or *Tetrafluoromethane.* See Carbon Tetrafluoride in Vol 2 of Encycl, p C64-L

"Freon–21". See Dichlorofluoromethane in this Section

"Freon–22" or *"F–22"* or *Monochlorodifluoromethane.* See chlorodifluoromethane in this Section

"Freon–113" or *"F–113"*. See Trichlorotrifluoroethane in this Section

"Freon–114" or *"F–114"*. See Dichlorotetrafluoroethane in this Section

"Freon–C318". See Octafluorocyclobutane in this Section

"Freon–BF", Solvent or *Tetrachlorodifluoroethane.* See 1,2-Difluoro-1,1,2,2-tetrachloroethane in this Section

"Freon–MF", Solvent or *Trichloromonofluoromethane.* See Fluorotrichloromethane in this Section

"Freon–TF", Solvent. See Trichlorotrifluoroethane in this Section

Ref: CondChemDict (1961), 515-L to 516-L and on pages indicated under their chemical names; (1971), 402-R

Hydrazine Tetrafluoride. See Tetrafluorohydrazine in this Section

Hydrofluoric Acid. See the first item described under FLUORINE DERIVATIVES in this Section

Hydrofluorosilicic (or Hydrofluosilicic) Acid. See Fluorosilicic Acid in this Section

Hydrogen Fluoride. Same as Hydrofluoric Acid, Anhydrous, described in this Section

Iodine Fluoride or **Iodine Pentafluoride,** IF_5, mw 221.91; colorless, fuming liq; sp gr 3.29 at 0° (Ref 3), 3.189 at 25° (Ref 3), fr p –8° (Ref 3), –9.4° (Ref 3), bp 97° (Ref 2), 98° (Ref 3); decompd by w & attacks glass. Can be prepd by igniting iodine in a stream of fluorine. Used as an incendiary agent & as fluorinating agent

Refs: 1) Gmelin-Kraut Syst Number 8 (1933), 601 2) Lange (1961), 258 3) CondChemDict (1961), 609-L; (1971), 472-R

Iron Fluoride. See Ferric Fluoride in this Section

Kalium Fluoride. See Potassium Fluoride in this Section

Lead Fluoride, PbF_2, mw 245.21; wh crysts, sp gr 8.24, mp 855°, bp 1290°; sl sol in w; sol in nitric acid; insol in NH_3 & acetone. Can be prepd from the elements, from reaction of F with Pb salts, and by reaction of HF with Pb salts (Ref 1). Used in electronic & optical applications

Refs: 1) Gmelin-Kraut Syst Number 47, Teil C (1969), 272 2) Lange (1961), 262 3) CondChemDict (1971), 509-L

Lithium Fluoride, LiF, mw 25.94; wh cu crysts, poisonous, sp gr 2.295 at 21.5°, mp 870°, bp 1670°; sl sol in w and does not react with it at red heat; insol in alc & acetone; sol in HF. Can be prepd by treating lithium carbonate with HF. Used in welding and soldering flux, in ceramics and for prepn of synthetic crysts in IR & UV instruments; proposed for use in space components

Refs: 1) Gmelin-Kraut Syst Number 20 (1927), 109; (1960), 305 2) CondChemDict (1961), 675-R; (1971), 524-L 3) Kirk & Othmer 9 (1966), 627

Magnesium Fluoride or *Magnesium Sellaite* (Magnesium Flux), MgF_2, mw 62.32, wh crysts; sp gr 3.0, mp 1263°, bp 2239°; exhibits fluorescence by elec light; insol in alc; nearly insol in w; sol in nitric acid. Can be prepd by adding hydrofluoric acid to Mg salt soln. Used in ceramics & glass

Refs: 1) Gmelin-Kraut Syst Number 27, Teil B (1939), 98 2) CondChemDict (1961), 690-R & 691-L; (1971), 535-L 3) Kirk & Othmer 9 (1966), 627

Magnesium Fluosilicate or **Magnesium Silicofluoride,** $MgSiF_6.6H_2O$; mw 274.51; wh, efflorescent crysts, sp gr 1.788 at 17.5°/4, mp – decomp on heating to 120°; sol in w. Can be prepd by treating Mg hydroxide or carbonate with hydrofluosilicic acid. Used in ceramics, concrete hardening, waterproofing & mothproofing
Refs: 1) Gmelin-Kraut Syst Number 27, Teil B(1939), 391 2) CondChemDict (1961), 691-L; (1971), 535-L 3) Kirk & Othmer **9** (1966), 660

Magnesium Flux. See Magnesium Fluoride in this Section

Manganic Fluoride, MnF_3, mw 111.94, red, poisonous crysts, sp gr 3.54; mp – decomp on heating; sol in acids; decomp by w. Can be prepd by dissolving Mn in dil HF contd in a silver basin (Ref 2) and by other methods. Used as a fluorinating agent
Refs: 1) Gmelin-Kraut, not found 2) Mellor **12**(1932), 342 3) CondChemDict (1961), 699-R; (1971), 542-L

Manganous Fluoride, MnF_2, mw 92.94, reddish pdr, sp gr 3.98, mp 8.56°; sol in acids; insol in w, alc & ether. Can be prepd by the action of HF on manganous hydroxide and by other methods (Ref 2)
Refs: 1) Gmelin-Kraut, not found 2) Mellor **12**(1932), 344 3) CondChemDict (1961), 700-L & R; (1971), 542-R

Mercuric (or Mercury) Fluoride, HgF_2, mw 238.61, transparent, poisonous crysts; sp gr 8.95 at 15°, mp 645°(dec); sol in HF & dil nitric acid; decomp by w. Can be prepd by treating mercuric oxide with HF. Used in synthesis of org fluorine compds
Refs: 1) Gmelin-Kraut Syst Number 34, Teil B(1967), 408 2) CondChemDict (1961), 714-R; (1971), 552-R 3) Kirk & Othmer **9** (1966), 628

Methyl Fluoride. See Fluoromethane in this Section

Molybdenum Fluoride or **Molybdenum Hexafluoride,** MoF_6, mw 209.95, wh crysts melting at 17.5°; sp gr of liq about 2.5; bp 35°; reacts readily with w. Can be prepd by direct action of fluorine on Mo metal. Used in the separation of Mo isotopes
Refs: 1) Gmelin-Kraut, not found 2) Mellor **11**(1931), 610 3) CondChemDict (1961), 761-R; (1971), 593-L

Natrium Fluoride. See Sodium Fluoride in this Section

Nickel Fluoride, NiF_2, mw 96.71, grn pdr, mp 1100°, sp gr 4.63; sl sol in w; insol in alc, eth & NH_3. Can be prepd from Ni carbonate & anhyd HF at elevated temp (Refs 1 & 3)
Refs: 1) Gmelin-Kraut Syst Number 57, Teil B (1966), 527 2) Lange (1961), 278 3) Kirk & Othmer **9** (1966), 628

Nitrogen Oxyfluoride. See Nitrosyl Fluoride in this Section

Nitrogen Trifluoride, NF_3, mw 71.01; colorless gas or mobile liquid; sp gr 1.537, fr p –206.6°, bp –128.8°. It is one of the *Nitrogen Fluorine Compounds.* Others are *Dinitrogen Tetrafluoride, Dinitrogen Difluoride* and *Difluoramine.* They are powerful oxidizers suitable for both liquid and solid rocket proplnts. Also used for high energy fuels
Refs: 1) Gmelin-Kraut Syst Number 5(1959), 238 2) CondChemDict (1961), 805-L; (1971), 623-L 3) Kirk & Othmer **9**(1966), 629

Nitrogen Trioxyfluoride. See Fluorine Nitrate in this Section

Nitrosyl Fluoride or **Nitrogen Oxyfluoride,** NOF, mw 49.01, colorless gas; sp gr 1.68

(Air=1), fr p −134°, bp −56°; decompd by w. Can be prepd by treating NaF with sodium nitrate & sulfuric acid (Ref 1)
Refs: 1) Gmelin-Kraut Syst Number 5 (1926), 51 2) Lange (1961), 280 (Nitrogen Oxyfluoride)

Nitroxyl Fluoride or **Nitryl Fluoride,** NO_2F, mw 65.01, colorless gas; sp gr 2.24 (Air=1), fr p −139°, bp −63.5°; decomp by w, alc & eth. Can be prepd by treating F with 2 moles of NO at liq O temp (Ref 1)
Refs: 1) Gmelin-Kraut Syst Number 5 (1926), 51 2) Lange (1961)

Nitryl Fluoride. See Nitroxyl Fluoride

Nitryl Hypofluorite. See Fluorine Nitrate in this Section

Octafluorocyclobutane ("Freon-C318"), $(CF_2)_4$, mw 200.04; chemically inert dry gas, bp −6° (760mm); high electric strength. Can be prepd by dimerization of tetrafluoroethylene at high temp. Used for electric equipment and as aerosol proplnt for foods and pharmaceuticals
Refs: 1) Beil **5**, [8] 2) CondChemDict (1961), 822-L & 515-R, under "FREON"; (1971), 638-L

Perfluoroethylene or **Tetrafluoroethylene.** See Ethylene Tetrafluoride in this Vol p E291-L

Peroxydisulfuryl Difluoride, $S_2O_6F_2$, mw 198.14; colorless liq having extremely unpleasant odor, fr p −55.4°, bp 67.1°; hydrolyzes in water. It ignites org materials immediately upon contact. It can be prepd by reaction of F with an excess of sulfur trioxide at 250°, and by combination of fluorine fluorosilicate with sulfur trioxide (Refs 3 & 4)
Refs: 1) Gmelin-Kraut Syst Number 5, Teil B (1963) 2) CondChemDict (1961), not found 3) F.B. Dudley, JChemSoc **1963**, 3407–11 & CA **59**, 2377 (1963) 4) J.M. Shreeve & G.H. Cady, InorgSynth **7** (1963), 124–28 & CA **61**, 269 (1964) 5) Anon, C&EN, Feb 21 (1966), p 40-R, under BRIEFS [During the prepn 500g of $S_2O_6F_2$ (the method is not indicated), about 200g of fluorine fluorosulfate (SO_3F_2) formed. Sulfuryl fluoride (SO_2F_2) also formed and possibly some unidentified substances. The by-products were distilled into a cold, clean, dry steel cylinder strong enough to withstand press of 135 atm. On warming to RT the cylinder violently exploded]

Phosphorous Pentafluoride, PF_5, mw 125.98, colorless gas, fr p −91.6°, bp −84.8°, d of gas 5.84g/liter; sol in or reacts with w, alcohols, ethers & amines; gas produces a fog in moist air; not corrosive to dry metals or glass, The toxicity is same order as that of BF_3. Phosphorous pentafluoride can be made by treating the chloride with arsenic trifluoride, and by thermal decompn of hexafluorophosphates & the diazonium salts. PF_3 may be converted to PF_5 by heating Cl alone, or with calcium fluoride
Refs: 1) Gmelin-Kraut Syst Number 16, Teil C (1965), 395 2) Kirk & Othmer **9** (1966), 635 3) Sax (1968), 1023 4) CondChem Dict (1971), 689-R

Phosphorous Trifluoride, PF_3, mw 87.98; colorless gas, fr p −151.5°, bp −101.8°, d of gas 3.91g/liter & d of liq at bp 1.6g/ml; almost insol in w; dissolves readily in alkaline solns; much more poisonous than PF_5 & POF_3. Excessive exposure may produce symptoms similar to those of CO. It can be prepd by fluorinating PCl_3 with CaF_2 or with AsF_3 or SbF_3
Refs: 1) Gmelin-Kraut Syst Number 16, Teil C (1965), 388 2) Lange (1961), 286 3) Kirk & Othmer **9** (1966), 636 4) Sax (1968), 1025 5) CondChemDict (1971), not found

Polyvinyl Fluoride. See Vinylfluoride Polymer in this Section

Potassium Bifluoride (Potassium Acid Fluoride), KHF_2, mw 78.11; colorless, poisonous & corrosive crysts; mp – decomp; sol in w & dil alc; insol in abs alc. Can be prepd by crystallizing from an aq soln of KF in HF (Ref 1). Used for etching glass; production of fluorine and of flux in metallurgy
Refs: 1) Gmelin-Kraut Syst Number 22(1937), 344 2) Lange (1961), 288 3) CondChemDict (1961), 925-R; (1971), 718-R

Potassium Borofluoride, Potassium Fluoroborate or **Potassium Fluoborate,** KBF_4, mw 125.92; gritty wh pdr (when dried), or gelatinous crysts of weak bitter taste; sp gr 2.5 at 20°, mp decomp 350°; bp – decomp; sl sol in w & hot alc. Prepd by mixing fluoboric acid with a soln of K hydroxide or carbonate; the resulting gelatinous ppt is washed and dried. Other method is heating of boric acid with K fluosilicate followed by adding K carbonate. Used in grinding wheels, in flux for silver soldering, in electrochemical processes and in chemical research
Refs: 1) Gmelin-Kraut Syst Number 22(1937), 818 2) CondChemDict (1961), 929-R & 930-L; (1971), 721-L 3) Kirk & Othmer, Vol **9**(1966), 564

Potassium Fluoborate. See Potassium Borofluoride

Potassium Fluoride: (a) KF, mw 58.10 and (b) $KF.2H_2O$, mw 94.13 (usual commercial form); wh poisonous crysts; sol in w & HF; insol in alc; sp gr 2.48 of (a) & 2.454 of (b); corresponding mp's 46° & 41°. Can be prepd by saturating hydrofluoric acid with K carbonate. Used for etching glass, as preservative and insecticide
Refs: 1) Gmelin-Kraut Syst Number 22(1936), 324 2) Lange (1961), 290 3) CondChemDict (1961), 930-L; (1971), 721-R 4) Kirk & Othmer **9**(1966), 649

Potassium Fluosililicate or **Potassium Silicofluoride** (Hieratite), K_2SiF_6, mw 220.29; wh crysts, sp gr about 3.0, mp–decomp; sl sol in w; insol in alc; sol in HCl. Can be prepd by reacting KCl with fluorosilicic acid. Used for prepg synthetic mica; in metallurgy of Al & Mg, ceramics & insecticides
Refs: 1) Gmelin-Kraut, not found 2) Lange (1961), 294 3) CondChemDict (1961),930-L; (1971), 721-R 4) Kirk & Othmer **9**(1966), 659

Potassium Fluozirconate. See Zirconium Potassium Fluoride in this Section

Rubidium Fluoride, RbF, mw 104.48; wh crysts, sp gr 2.88 at 820°, mp 775°, bp 1410°; v sol in w, sol in dil HF, insol in eth & NH_3. Can be prepd by heating Rb hydroxide or carbonate with HF in a Pt dish and by other methods (Ref 1)
Refs: 1) Gmelin-Kraut Syst Number 24(1937), 125 2) Lange (1961), 296 3) CondChemDict (1971), 768-L

Selenium Tetrafluoride, SeF_4, mw 154.96, colorless liquid, sp gr 2.77 at 20°, fr p –80°, bp +100°; decompd by w. Can be prepd by action of F on selenium in a Cu vessel, or by the action of dry Ag fluoride on Se tetrachloride (Ref 2)
Refs: 1) Gmelin-Kraut, not found 2) Mellor **10**(1930), 893 3) Lange (1961), 300

Silicofluoric Acid. See Fluorosilicic Acid in this Section

Silicofluoroform, $SiHF_3$, mw 86.10, colorless gas, sp gr 2.98° at 0°(Air=1), fr p –110°, bp –80.2°; dec by w, alc, eth & alkalies; sol in toluene. Can be prepd by action of AsF_3, SnF_4 or TiF_4 with $SiHCl_3$ (Ref 1)
Refs: 1) Gmelin-Kraut Syst Number 15, Teil B(1959), 636 2) Lange (1961), 300

Silicon Fluoride or **Silicon Tetrafluoride,** SiF_4, mw 104.09; colorless, nonflammable gas with suffocating odor similar to HCl; fumes strongly in air, sp gr 3.57 (Air=1); fr p –90°, bp –86°; absorbed in large quantity of w with decompn; sol in alc, eth &

nitric acid. Can be prepd either by action of HF on silica or by direct synthesis. Used in manuf of fluosilicic acid and in chem analysis
Refs: 1) Gmelin-Kraut Syst Number 15, Teil B(1959), 614 2) Lange (1961), 302 3) CondChemDict (1961), 1020-R; (1971), 785-L

Silver Fluoride, AgF, mw 126.88, yel or brn crysts; sp gr 5.852 at 15.5°, mp 435°, bp 1159°; sol in w, sl sol in NH_3; very hygroscopic; forms hydrate $AgF.H_2O$; becomes dark on exposure to light. Can be prepd by heating N. Used in medicine
Refs: 1) Gmelin-Kraut Syst Number 61, Teil B (1971), 293 2) Lange (1961), 304 3) CondChemDict (1961), 1022-R; (1971), 787-L 4) Kirk & Othmer **9**(1966), 661

Sodium Borofluoride or **Sodium Fluoroborate,** $NaBF_4$, mw 109.81; wh cryst pdr with bitter taste; sp gr 2.47 at 20°; sp 384° with slow decompn; sol in w, sl sol in alk. Can be prepd by heating NaF & hydrofluoboric acid, followed by slow cooling & crystn. Used as fluorinating agent, also in fluxes for nonferrous metals, in electrochem processes and as oxidation inhibitor
Refs: 1) Gmelin-Kraut Syst Number 21 (1928), 681 2) Lange (1961), 308 (Fluoborate 3) CondChemDict (1961), 1041-R; (1971), 800-R

Sodium Fluoride (Villaumite), NaF, mw 41.99; colorless, poisonous crysts, avoid breathing dust; sp gr 2.766, mp 993°, bp 1704°; sol in w; sl sol in alc. Can be prepd by treating Na carbonate with HF. Used for fluorination of municipal water supply, in insecticides, fungicides, in metallurgy (rimmed steel), electroplating, manuf of glass and vitreous enamels; single crysts used as windows in UV & IR radiation detecting systems. May be fatal if swallowed
Refs: 1) Gmelin-Kraut Syst Number 21 (1928), 297 2) Lange (1961), 308 3) CondChemDict (1961), 1041-R; (1971), 800-R

Sodium Fluorosilicate or **Sodium Silicofluoride** (Salufer), Na_2SiF_6, mw 188.07; wh, odorless, poisonous, granular pdr; sp gr 2.679; mp–decomp at red heat; v sl sol in cold w; better sol in hot w; insol in alc. Can be prepd by neutralization of fluosilicic acid with Na carbonate. Used for fluoridation of drinking w; for manuf opalescent glass, latex foam rubber, metallurgy of Al & Be, insecticide, chem intermediate, etc
Refs: 1) Gmelin-Kraut Syst Number 21 (1928), 882 2) Lange (1961), 310 3) CondChemDict (1961), 1042-L; (1971), 801-L

Stannic Fluoride, SnF_4, mw 194.70, wh crysts, sp gr 4.78 at 19°/4, mp 705°; v sol in cold w; decompd by hot w. Can be prepd by fluorinating Sn with ClF_3, or by reaction of NOF.3HF x ith metallic Sn in liq phase at 20–120° (Ref 1), and by reacting SnF_2 with F (Ref 3). Used in industrial applications as a catalyst
Refs: 1) Gmelin-Kraut Syst Number 46, Teil C(1972), 171 2) Lange (1961), 312 3) Kirk & Othmer **9**(1966), 682

Stannous Fluoride or **Tin Fluoride,** SnF_2, mw 156.72, wh crysts, mp 210–215°; bp–decompd in air to $SnOF_2$; sl sol in w; slowly hydrolyzes. Can be prepd by the dissolution of stannous oxide in aq HF or by dissolving metallic Sn in anhyd or aq HF (Refs 1 & 3). Used in toothpaste as fluoride source
Refs: 1) Gmelin-Kraut Syst Number 46, Teil C(1972), 154 2) CondChemDict (1961), 1070-R; (1971), 823-L 3) Kirk & Othmer **9**(1966), 682

Strontium Fluoride, SrF_2, mw 125.63; wh pdr, sp gr 4.22, mp 1400° (Lange), 1190° (Ref 2); nearly insol in w; sol in hot HCl. Can be prepd by treating an aq $SrCl_2$ soln with Na or K fluoride (Ref 1). Used as substitute for other fluorides
Refs: 1) Gmelin-Kraut Syst Number 29 (1960), 187 2) CondChemDict (1961), 1080-R; (1971), 829-R

Sulfur Hexafluoride, SF_6, mw 146.071 colorless gas; sp gr 5.11 at 20°(Air=1), of liq 1.79 at −39°, fr p −50.8°(under pressure), bp−sublimes at −63.5°; v.sl sol in w; sol in alc, eth & KOH. Prepd by interaction of sulfur & fluorine. Used as dielectric (gaseous insulator for electrical equipment)
Refs: 1) Gmelin-Kraut Syst Number 9, Teil B(1963), 1711 2) Lange (1961), 316 3) CondChemDict (1961), 1093-L; (1971), 837-L

Sulfuric Ocyfluoride or **Sulfuryl Fluoride,** SO_2F_2, mw 102.07, colorless gas, fr p −136.7°, bp −55.4°; sol in w & alk; insol in concd sulfuric acid. Can be prepd by heating an excess of F & S in a glass tube with SF_6 & SO_2F_2 added, or by heating gaseous F & SO_2 & SO_2F_2 (Ref 1). Used as insecticide & fumigant
Refs: 1) Gmelin-Kraut Syst Number 9, Teil B (1963), 1729 2) Lange (1961), 316 3) CondChemDict (1971), 838-R

Sulfurous Oxyfluoride, SOF_2, mw 86.07, colorless gas, fr p −110°, bp −30°; sol in benz & ether; decompd by w. Can be prepd by heating F with S in a glass tube and in the presence of the oxyfluoride of sulfur (Ref 1), and by other methods
Refs: 1) Gmelin-Kraut Syst Number 9, Teil B (1963), 1724 2) Lange (1961), 318 3) CondChemDict (1961), not found

Tetrafluorodichloroethane. See Dichlorotetrafluoroethane in this Section

Tetrafluoroethylene or **Perfluoroethylene.** See Ethylene Tetrafluoride in this Section

Tetrafluorohydrazine or **Hydrazine Tetrafluoride,** $F_2N.NF_2$, mw 104.02; colorless, mobile liq or gas, bp(calcd) −73°; crit temp +36°, heat of vaporization 3170cal/mole. Can be prepd by reaction of nitrogen trifluoride with metals at high temp (Ref 3). Explodes in contact with reducing agents. Used as oxidizer in fuels for rockets or missiles; also in org synthesis
Refs: 1) Gmelin-Kraut, not found 2) CondChemDict (1961), 1122-L; (1971), 857-R 3) Kirk & Othmer 9(1966), 629

Tetrafluoromethane or **"Freon-14".** See Carbon Tetrafluoride in Vol 2, p C64-L

Thorium Tetrafluoride, ThF_2, mw of anhydrous 308.05; wh pdr having approx formula $ThF_4 . 1.4H_2O$; dehydrated betw 200° & 300°; mp 1111°; reacts above 500° with atm moisture to form oxyfluoride $ThOF_2$ and finally the oxide ThO_2. Forms a series of compds with other metallic fluorides, such as KF & NaF. Thorium tetrafluoride can be prepd by action of F on thorium chloride or bromide at RT (Ref 1). Used for prepn of Th metal and Mf−Th alloys; also in high temp ceramics
$ThOF_2$ is used as a protective coating on reflective surfaces
Refs: 1) Gmelin-Kraut Syst Number 44(1955), 252 2) Lange(1961), 320 3) CondChemDict(1961), 1139-L; (1971), 870-R

Tin Fluoride. See Stannous Fluoride in this Section

Titanium Tetrafluoride, TiF_4, mw 123.90; wh pdr, sp gr 2.798 at 20.5°, mp (?), bp 284°; sol in w, alc & pyridine; insol in eth. Can be prepd by reacting Ti with F_2 at 250° or thru reaction of $TiCl_4$ with anhyd HF. Used Used in catalytic application and in the formation of adducts
Refs: 1) Gmelin-Kraut Syst Number 41(1951), 287 2) Lange(1961), 322 3) Kirk & Othmer 9(1966), 683 4) Sax (1968), 1167-R

Titanium Trifluoride, TiF_3, mw 104.90; viol-red pdr; sp gr 2.98; on heating in air it is transformed slowly to TiO_2; sol in cold w; decompd by hot w. Can be prepd by passing HF over heated Ti hydride or by

reacting HF with $TiCl_3$. Used in catalytic applications
Refs: 1) Gmelin-Kraut Syst Number 41 (1951), 287 2) Lange (1961), 322 3) Kirk & Othmer **9** (1966), 683 4) Sax (1968), 1168-L

Trichlorofluoromethane. See Fluorotrichloromethane ("Freon-11") in this Section

1,1,2-Trichloro-1,2,2-trifluoroethane or **Trifluorotrichloroethane** ("Freon-113" or "Freon-TF"), $FCl_2C.CClF_2$; mw 187.39; colorless, nearly odorless, vol liq; sp gr 1.576 at 20°, fr p −35°, bp 47.6°; insol in w; miscible with alc, eth & benz. Can be prepd from perchloroethylene & hydrofluoric acid. Used as solvent, refrigerant, fire extinguisher and for prepn of chlorotrifluoroethylene
Refs: 1) Beil **1**, {157} & <142> 2) CondChemDict (1961), 1163-L and 515-R & 516-L, under FREON; (1971), 888-R

Trifluorochloroethane. See 1-Chloro-1,2,2-trifluoroethane in this Section

Trifluorochloroethylene. See 2-Chloro-1,1,2-trifluoroethylene and its polymer, Fluorothene, in this Section

Trifluorochloromethane. See Chlorotrifluoromethane in this Section

Trifluorochloromethylbenzenes. See Chlorobenzotrifluorides in this Section

Trifluorochlorotoluene. See under Chlorobenzotrifluorides in this Section

Trifluoromethane. See Fluoroform in this Section

Trifluoromethylbenzene. See Benzotrifluoride in this Section

Trifluoronitrosomethane, CF_3NO, mw 99.02; bright blue, fairly stable gas, very irritant; fr p −150°, bp −84°. Can be prepd by interaction of iodo (or bromo) trifluoromethane with nitric oxide in the presence of UV light. Serves as a monomer for nitroso rubber & as a military poison
Refs: 1) Beil **1**, {105} & <99> 2) CondChemDict (1961), 1167-R; (1971), 893-L

Trifluorotrichloroethane. See Trichlorotrifluoroethane in this Section

Tungsten (or Wolfram) Hexafluoride, WF_6, mw 297.86; colorless gas or lt yel liquid; toxic; sp gr (liq) 3.44, fr p 2.5°, bp 19.5°. Can be prepd by direct fluorination of powdered tungsten, followed by distillation under pressure. Used for vapor phase deposition of tungsten and as fluorinating agent
Refs: 1) Gmelin-Kraut Syst Number 54 (1933), 155 2) Lange (1961), 324 3) CondChemDict (1961), 1180-L; (1971), 905-R

Uranium Hexafluoride, UF_6, mw 357.07; colorless, volatile crysts; sublimes; sp gr 4.68 at 21° (Ref 1), 5.06 at 35° (Ref 2), mp 69.2° at 2 atm (Ref 1), 64.5° at 2 atm (Ref 2); triple p 64.0° at 1134mm (Ref 2); bp 56.2° at 765mm (Ref 1); sol in cold w (Ref 1); reacts vigorously with w, alc, eth and most metals (Ref 2). Obtd by fluorination of U tetrafluoride (qv). Used in gas diffusion process for separating isotopes of U
Refs: 1) Gmelin-Kraut Syst Number 55 (1936), 121 2) Lange (1961), 324 3) CondChemDict (1961), 1193-L; (1971), 914-L

Uranium Tetrafluoride (Green Salt), UF_4, mw 314.07; grn, non volatile pdr; sp gr 6.70; mp 1000° (Ref 2), 1036° (Ref 3); bp 1417° (Ref 2); insol in w & dil acids; sol in concd acids. Prepd by hydrofluorination of UO_2, which, in turn, is obtd from fluid bed reduction of higher oxides by hydrogen. Used for prepn of U metal and UF_6
Refs: 1) Gmelin-Kraut Syst Number 55 (1936),

120 2) Lange(1961), 324 3) CondChem-Dict(1961), 1193-R; (1971), 914-R

Vanadium Pentafluoride, VF_5, mw 145.95; colorless or yel pdr, sp gr 2.177 at 19°, mp ?, bp 111.2°; sol in w, alc & chlf; insol in CS_2. Can be prepd by action of F on metallic V or vanadium chloride (Ref 1)
Refs: 1) Gmelin-Kraut Syst Number 48, Teil B(1967), 187 2) Lange(1961), 326 3) Sax(1968), 1224-L

Vanadium Trifluoride, VF_3 & $VF_3.3H_2O$. See Lange (1961), p 326; Sax(1968), 1224-R and Gmelin-Kraut Syst Number 48, Teil B (1967), 181

Vinyl Fluoride. See Fluoroethylene in this Section

Vinylidene Fluoride. See 1,1-Difluoroethylene in this Section

Vinylidene Resins (Polyvinylidene Resins). These are resins in which the unit structure in the polymer molecule is $(-H_2C.CX_2-)$. Here the X usually stands for F, Cl or CN radical. Examples are "Saran", "Viton A" and "Gene-1132A"
Refs: 1) CondChemDict(1961), 1212-L; (1971), 928-R 2) Kirk & Othmer **21**(1970), pp 269–303; "Vonylidene Polymers" by R. Wessling & F.G. Edwards

Vinylfluoride Polymer (Polyvinyl Fluoride), $(-H_2C.CHF-)_n$. A transparent solid used for prepn of films. It possesses superior resistance to chemicals and outdoor weather. The film is flexible, tough, has high dielectric constant, has low permeability to air & water and has high resistance to heat. This polymer may serve as replacement of paints and as an outdoor protective coating for siding & roof; also for glazing, packaging, and electrical uses
Refs: 1) CondChemDict(1961), 921-R; (1971), 714-R 2) Kirk & Othmer **9**(1966), pp 835–40; "Polyvinyl Fluoride" by L.E. Wolinski

Zinc Fluoride, ZnF_2, mw 103.38; wh, poisonous pdr; sp gr 4.84 at 15°, mp 872°, bp ca 1500°; sol in hot acids, sl sol in w, insol in alc. Can be prepd by the action of HF on Zn hydroxide or by the addn of NaF to a soln of Zn acetate. Used in ceramic glazes & enamels; impregnating lumber and galvanizing
Its tetrahydrate, $ZnF_2.4H_2O$, is described in Ref 2, p 830
Refs: 1) Gmelin-Kraut Syst Number 32(1956), 844 2) Lange(1961), 330 3) CondChem-Dict(1961), 1245-R; (1971), 950-L

Zinc Fluorosilicate or **Zinc Silicofluoride,** $ZnSiF_6.6H_2O$; mw 315.57; wh crysts, sp gr 2.104 at 20°, mp–dec; sol in w. Can be prepd by treating Zn oxide with fluosilicic acid. Used for concrete hardener and as preservative & mothproofing agent
Refs: 1) Gmelin-Kraut Syst Number 32(1956), 993 2) Lange(1961), 330 3) CondChem-Dict(1961), 1249-L; (1971), 950-L 4) Kirk & Othmer **9**(1966), 660

Zirconium Fluoride (Zirconium Tetrafluoride), ZrF_4, mw 167.22; wh crysts, sp gr 4.43, mp 600° (subl); sl sol in cold w; decompd by hot w; sol in HF. Can be prepd by treating $ZrCl_4$ with anhyd HF or by thermal decompn of $(NH_4)_3ZrF_7$ which can be obtd by reacting ZrO_2 with NH_4HF_2 (Refs 1 & 3). Used as a component of molten salts for nuclear reactors
Refs: 1) Gmelin-Kraut Syst Number 42(1958), 278 2) Lange(1961), 332 3) Kirk & Othmer **9**(1966), 685 4) CondChemDict (1971), 957-R

Zirconium Potassium Fluoride or **Potassium Fluozirconate,** K_2ZrF_6, mw 283.42; wh crysts, sp gr 3.58; sol in hot w, insol in NH_3. Can be prepd by treating a KF soln with an excess of ZrF_4 soln (Ref 1). Used in prepn of metallic Zr

Refs: 1) Gmelin-Kraut Syst Number 42(1958), 412 2) Lange(1961), 290(Potassiumfluozirconate) 3) CondChemDict(1961), 1254-R; (1971), 957-L 4) Kirk & Othmer **9**(1966), 685

Flüssige Tri (Ger & Swiss). Liquid TNT. See 2,4,6-Trinitrotoluene under Toluene

Fluted Liners. In the development of shaped-chge projectiles it became known that spin reduced the penetration capabilities of shell with smooth liners. To obtain better penetration with a spinning chge, so-called *fluted liners* were developed wherein the exterior surface had ridges emanating from the apex. Initial ballistic tests of such liners made by the rubber-covered-punch process and adapted for assembly into modified 57mm M307A1 HEAT Shell gave promising results

Shells (loaded with Comp B) with these liners were tested by firing from the M18 rifle. These shells which rotated at about 210rpm, penetrated at least 3 inches in armor-plate in 96% of trials and 6 inches in 40% of trials. When fired at 120rpm in a spin-test app, the average penetration in mild steel was 7.4 inches. The standard 57mm M307A1 HEAT Shell which contains a hemispherical liner cannot be relied upon to penetrate 3 inches of armor plate

Further development work on liners made by the rubber-covered-punch process was deferred pending evaluation of liners made by the *matched-die* process when studies indicated that the latter process should produce liners of greater uniformity (Ref 4)

Although the effects of rotation were not known with certainty, it was theorized that when the liner collapses in a rotating shaped chge, the jet formed from the liner also rotates and spreads or splits. To counteract these effects of rotation, tangential components of velocity are necesssry. These must be equal in magnitude but opposite in direction to those set up by the initial spin upon all the elements of the liner. The purpose of this counteracting force, called *"spin-compensation"*, is to stop rotation of the jet (Ref 1). Both theory and initial test results indicated that a fluted liner offered the best means of compensating for rotation (Ref 2)

Such a liner was designed in 1946 at the Carnegie Inst of Techn (CIT) and the Nat Bur of Standards (NBS) started work to develop a method for manufg such liners

The rubber-covered punch process is described in Ref 3

Refs: 1) W.T. August et al, "The Effect of Rotation Upon Shaped Charge Jets", Chap VII, BRL Rept **905**, May 1954(Project TB3-0134) 2) Staff, Carnegie Inst of Tech, Pittsburgh, Pa, Bimonthly Reports under Contracts Nos W-36-06-ORD-2879, DA-36-061-ORD-7, DA-36-061-ORD-2 and DA-36-061-ORD-291 3) Staff, Camin Labs Inc, Brooklyn, NY, Progress Reports under Contract No DA1-28-017-ORD-(P)-488 4) G.D. Clift, "Perfomance of 57mm Shell Containing Fluted Liners Coined by the Rubber-Covered-Punch Process", PATR **2293**(1956)

Fly Cotton. It consists mostly of dead or unripe cotton fibers from plants which have not reached maturity as a consequence of attacks by parasites, genetic factors or lack of nutriment. The fibers collect into tangled masses called *"neps"*. Dead fibers may be recognized in cotton from the fact that they are doubly refractive, showing colors under a polarized microscope. If a mica plate is inserted, the normal (ripe) fibers remain bright in all positions, while the dead fibers show black and white portions (according to their direction)

Unripe fibers show less contrast than dead ones under the polarized microscope with mica plate. The unripe fibers are of about the same width as the normal fibers but are not twisted, while "dead" fibers are broad and flat

Both dead and unripe fibers are not suitable for nitration because they produce unstable NC

Refs: 1) Marshall **1**(1917), 167 2) Ibid, **2** (1917), 696 3) Ibid(1932), 33

Flying Spot Microscope. A television-type scanner illuminates specimen in compound microscope to obtn pictures superior in size, brightness, contrast & resolution to those of normal microscope. Discrete particles are automatically counted and sized
Ref: F. Roberts, J.Z. Young & D. Causley, Electronics **26**, 137–39 (July 1953)

FNH Propellants. Flashless-nonhygroscopic propellants were developed after WWI in the US by incorporating into ordinary smokeless proplnts ingredients such as DNT, TNT, DPhA, DBuPh, hydrocellulose etc (See Table F8)

Table F8

Ingredients	Single-Base Propellants			Double-Base Propellant
	Formulation 1	Formulation 2	Formulation 3	
NC (12.6% N)	74	85	–	–
NC (13.25% N)	20		–	–
NC (13.0% N)	–	–	76–79	49
NG	–	–	–	20
DNT	5	10	21–24	–
TNT	–	–	–	20
DBuPh	–	5	–	–
DPhA	1	1(added)	0.2–1.2	1
Hydrocellulose	–	–	–	10

The term "flashless" is relative and depends on the caliber of the weapon in which it is used and, therefore, on the size of the charge. The compositions given above are nearly flashless proplnts for small-caliber guns, incl 75mm, but they would not be flashless in larger guns. For these it is necessary to add either more coolants, such as DBuPh or about 1% or more K sulfate or other salts (See also Cannon Propellant, p C29 in Vol **2** of Encycl and Flash Reducing Agents in this Vol)
Refs: 1) D.R. Cameron, PATR **10**(1931) 2) Hayes (1938), 8, 14 3) Davis (1943), 327 4) Ohart (1946), 26–7 5) Fedoroff & Clift **4**(1946), 36

Foamed Explosives for Floating Mine. An invention by Stark (Ref 1) provides a floating mine which consists of an expl having a closed-cell foam structure of greater and more shattering effect than the same wt of high-density expl. Buoyancies of these expls are of 20 to 50 lbs per cu ft. The foamed expls consists of HE's (such as TNT, NC, PETN or RDX) bonded by a thermosetting resin
Ref: H.J. Stark, USP 2845025 (1958) & Ordn **43**, 662 (Jan-Feb 1959)

Foams. See under Emulsification, Emulsion, Foaming and Foam in Vol **5**, p E87-R
Addnl Ref: J.J. Bickerman et al, "Foams. Theory and Application", Reinhold, NY (1953)

FOBS (Fractional Orbital Aerospace Bombardment System). A statement by Secretary of Defense, Robert S. McNamara, to the press on 3 November 1967 acknowledging the development by the Soviets of a FOBS. McNamara claimed in his pronouncement that FOBS posed no critical new threat
Refs: 1) Time, Vol **90**, No 19, p 25 (10 Nov 1967) 2) Staff, Ordn **52**, 354 (Jan-Feb 1968)

Fog. Any heterogeneous suspension of droplets of water or other liquid substances in a gas which disturbs its transparency may be called a fog. If the particles in suspension are solid, the fog is usually called smoke or "smog". In Chemical Warfare Service, the term "Smoke" is used for both liquid and solid suspensions
Refs: 1) Hackh's Dict(1944), 349 2) Vol 2 of Encycl, "CHEMICAL AGENTS", pp C165-R to C171-R

Fog Acid.(Nebelsäure in Ger). A smoke-screen agent consisting of 50/50–Chlorosulfonic acid/Sulfur trioxide (by weight)
Refs: 1) R.E. Richardson et al, CIOS Rept **15–18**(1945), p 6 2) PATR **2510**(1958), p Ger 116-R (Nebelsäure)

Fog Point. The temperature at the point when a clear liquid becomes opaque is called the fog point
Ref: R.D. Miller, Analyst **74**, 652(1949)

Fog Signals. See under Signals

Föhn Gerät or RZ Föhn (RZ 73 Flight). A 73mm air-to-air missile developed in 1941 by converting a Ger Army rocket. It used solid propInt and could be considered as predecessor of **R–4/M.** This was a 2-inch solid propInt rocket which carried about 1 pound of HE and had tail surfaces which could be folded back. It was mass-produced towards the end of WWII at the Deutsche Waffen- und Munitionsfabriken at Lübeck. As many as 48 of these missiles could be carried on the underwing racks of a fighter plane and fired practically simultaneously against a bomber formation at a range of 1200 to 1500 yards. It was claimed that a single hit with such a rocket was sufficient to bring down a bomber (Ref 1)

Accdg to Gatland (Ref 2), missile **R4/M** had diam 2.16 inches, overall length 2.75 ft, launching weight 7.75 lbs, and range 0.5 mile. It used a singular tubular grain propInt which had a burning time of 0.8 sec
Refs: 1) W. Dornberger, "V–2", Viking Press, NY(1954), 270 2) K.W. Gatland, "Development of Guided Missile", "Flight" Publication, London(1952), 122 3) PATR **2510**(1958); pp Ger 158 & 170

Folded Head Ammunition. One of the earlier American "center-fire" systems, used from about late sixties to about eighties of the last century. It consisted of straight cylindrical copper cartridge, which looked on the outside like the present *"rim-fire"* cartridge. As a primer it had a copper cup (contg some fulminating mixture) of diam slightly smaller than the inside of cartridge. The end of the cartridge and
than the inside of cartridge. The cup was inserted from the front end of the cartridge and pressed until it touched the bottom of the cartridge. After this the charge of propInt (BkPdr) was added and finally the bullet inserted. On firing, a hammer, or a firing pin crushed the flat copper head at the center which ignited the primer composition
Ref: M.M. Johnson & C.T. Haven, "Ammunition: Its History, Development and Use, 1600–1943", W. Morrow (1943), p 43

Folded Head Cartridge. See Vol 4, p D756-L

Folding Skirt Projectile (Tapered Bore). This type of projectile is fired thru a tapered bore which may be either built into the gun or added to a std gun by a special muzzle attachment. The taper serves to swage down the flanged skirts which extend from the main body of proj as a jacket. As a result the emergent caliber is much less than the original diam. In this way, the accelerating pressure of the powd gases acts on a large area in the gun bore, while a small (emergent) area is presented to the resisting pressure caused

by air resistance in flight

This type of proj has, therefore, the advantage of good exterior ballistics from the standpoint of ballistic coefficient. However, the swaged down skirts make imperfect contact with the proj body, thus creating generating points for retarding shock waves at high velocities

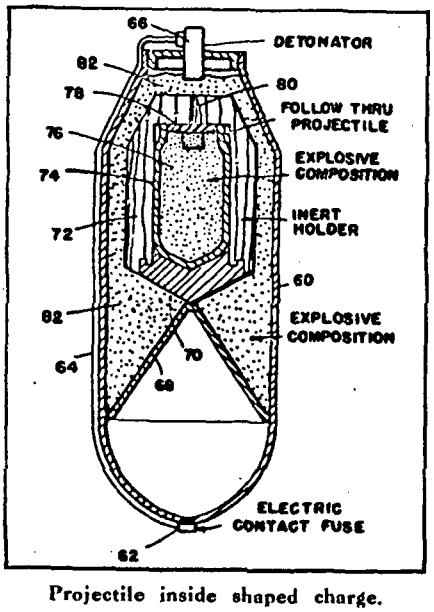
There are two serious disadvantages of the folding skirt projectile. The taper prohibits the use of std ammo in tapered bore guns and also increases tremendously the time & costs of producing both gun & ammo. In addn the tube is short lived. The Germans made use of this principle, however, in some of their weapons

Ref: Anon, Armament Engineering (1954), p 209

Follow-Through Projectile for Shaped Charges. The present method of obtaining efficiency from a shaped chge is to have the length of the expl chge behind the cavity liner equal to two or three times the diam of the cavity liner. Using this method, any follow-through projectile must be placed behind the point of deton. No successful proj of this type has yet been presented because of the difficulty of causing the proj to travel thru the deton products and enter the hole in target where jet penetrated. The object of this invention is to increase the efficiency of shaped chges by introducing a follow-thru proj or corrosive agent in the jet hole formed by the chge

In the follow-thru proj (See Fig), the deton of the expl causes the deton wave to pass around the inert core and to impact with the liner which sends out a narrow jet, piercing the target. In the meantime, the proplnt behind the follow-thru proj has burned, propelling the proj thru the jet hole to explode inside the target

Ref: S.A. Moses, USP 2809585 (1957) & Ordn **43**, 130 (July–Aug 1958)



Projectile inside shaped charge.

Fonberg Effect. See Fonberg Phenomenon

Fonberg Phenomenon. Z. Fonberg (Ref 1) observed that the deton of shaped charges of certain nitrocompds with lined cavity is accompanied by the appearance of electromagnetic radiation which seems to be of such intensity that evidence of nuclear transmutation has been observed. These experiments of Fonberg were repeated at la Poudrerie de Vonges (France) and described by Tauzin (Ref 2). No definite conclusion can be drawn from these investigations and further work is required if it is desirable to know whether the phenomenon is due to transmutation, to radiation, or to other cause

Refs: 1) Z. Fonberg, JChemPhys **19**, 383 (1951) 2) P. Tauzin, MAF **26**, 493–96 (1952) 3) G.J. Plain et al, JChemPhys **20**, 1049 (1952) [Translated in MAF **27**, 239–40 (1953)] 4) P. Tauzin, MAF **27**, 241–42 (1953) 5) Maj W.H. Gross, Explosivst **1956**, 169–74, 201–06 (20 refs) 6) H. Fiene, Explosivst **5**, 36–37 (1957)

Fonckite V. A mining expl which contd: Amm Perchlorate 53.24, Amm Oxalate 32.30 & TNNaphthalene 14.46%

Ref: Gody (1907), 712

Fonction de progressivité expérimentale (Fr for Function of Experimental Progressivity). In order to determine the so-called "vivacité expérimentale des poudres" (experimental quickness of propellants) from the data obtained when proplnts are fired in a manometric bomb, it is necessary first to determine the so-called "fonction de progressivité". For description of this function, see the following publications:
1) J. Dupuis, MAF **17**, 799–830(1938)(Note on the laws of combustion of proplnts)
2) J. Dupuis & M. Calvet, MAF **18**, 37–66 (1939)(Tests in the Drupp-Schmidt bomb)
3) M. Prache, MP **32**, 343–375(1950)(Contribution to the study of the "fonction de progressivité expérimentale des poudres" on firing in a manometric bomb; application to the examination of surface-treated proplnts

Fontaine Powders. These powders were fabricated by Fontaine before 1869 right in the center of city of Paris, near Sorbonne. They contained Potassium Picrate and Potassium Chlorate and were used for a while for fill ng shells and torpedoes. They were very powerful and superior to Designolle powders. However, they were very sensitive and their manufacture was stopped after a disastrous explosion occurred in 1869 at the plant, killing five workers (1892)
Refs: 1) Cundell (1889) in MP **5**, 336(1892) 2) Daniel (1902), 616(Under Picrates) 3) Clift & Fedoroff **2**(1943), p F2 4) Guia, Trattato **VI**(1), (1959), 397

Fontano, Barelly & de Choisy Powder. Military and sporting proplnts proposed in 1869: a) K chlorate 70.4 & red lead 29.6% b) K ferrocyanide 42.5, sugar 29.3, carbon 13.2, K permanganate 8.8, hydrocarbon 2.9, licopodium 1.7 & powdered gum 1.5%
Ref: Giua, Trattato **VI**(1)(1959), 392

Fontana, Nicola (called Tartaglia)(1500–1557). An Italian scientist, the creator of theory of artillery. The word "artillery" was first used by him, deriving it probably from "arte de tirare"
Ref: 1) A.D. Blinov, KursArtillerii, Voynnoye-Izdatel'stvo, Moscow, V **1** (1948)

Forbidden Explosives. According to Sec 73.51 of ICC's (Interstate Commerce Commission's) Regulations, the offering of the following explosives for transportation by common carriers by rail freight, rail express, highways, or water is forbidden:

1) Explosive compns that ignite spontaneously or undergo marked decompn when subjected for 48 consecutive hrs to a temp of 75°(167°F)
2) Expl compns contg an Amm salt and a chlorate
3) Liquid NG, DEGDN or other liq expls not defined as acceptable expls
4) Expls condemned by the Bureau of Explosives
5) Leaking or damaged packages of expls
6) Condemned or leaking Dynamite unless it was repacked by a competent person in the presence of, or with the written consent of, an inspector, or with the written authority of the Chief Inspector of the Bureau of Explosives
7) Firecrackers, flash crackers or salutes, the explosive content of which exceeds 12 grains in weight each, or pest control bombs, the expl content of which exceeds 18 grains each in weight
8) Fireworks that combine an explosive and a detonator or blasting cap
9) Fireworks contg an Amm salt and a chlorate
10) Fireworks contg yellow or white phosphorus
11) Fireworks or fireworks compns which behave as described in (1)
12) Fireworks condemned by the Bureau of Explosives, except when properly repacked as samples for lab examinations
13) Toy torpedoes, the maximum outside dimension of which exceeds 7/8 inch, or toy torpedoes contg a mixture of K chlorate, black antimony and sulfur, with an average wt of mixture in each torpedo exceeding 4 grains

14) Toy torpedoes contg a cap composed of a mixture of red phosphorus and K chlorate exceeding an average of 0.5 grains per cap
15) Fireworks contg Cu sulfate and a chlorate
16) New expls (except samples for lab examination) until approved for transportation by the Bureau of Explosives
17) Loaded firearms

Refs: 1) Agent H.A. Campbell's Tariff No 10, "Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles by Land and Water", 30 Vesey St., New York 7, NY (1957) 2) R.M. Graziano's Tariff No 27, "Hazardous Materials Regulations of the Department of Transportation", Association of American Railroads, Washington, DC 20036

Force and Covolume of Colloidal Propellants. Pressure-density of loading relationships were detd in France for various chem compns and grain forms. Suitable corrections were made for cooling effects by the walls of bombs. The data were used as a basis for calculating force and covolume. Theory and exptl values agreed satisfactorily

Ref: H. Muraour & G. Aunis, MAF **22**, 133–70 (1948) & CA **44**, 8659 (1950)

Force and Impetus in Detonation. See Vol **4**, pp D391-R & D393-L under Detonation (and Explosion), Impetus and Available Energy and also Dunkle's Syllabus (1957–1958), p257

Force Brisante. Fr for Bursting Power

Force Constant. The decompn of a unit mass of proplnt always liberates the same amt of energy which then heats the product gases to the same temp independent of density. For most proplnts, the most important equilibrium is the water gas equilibrium and, since this is equimolar, the assumed equation of state is sufficiently accurate for use in the interior ballistic of guns

It is std practice in the formulation of interior ballistic theory to assume an equation of state of the simple covolume type. For a gas obeying the Abel equation of state, the internal energy depends only on temp and not on density. It is expressed as

$$P(V-\eta) = nRT$$

In interior ballistics it is usually written in terms of a unit wt of gas, so that V & η have dimensions vol per unit wt, and n is the number of moles per unit wt. Many authors also define R as the gas constant per unit wt so that n does not appear explicitly in the equation. R so defined is not constant unless n is also constant

If T_o is the adiabatic flame temp, the energy released by the decompn of unit wt of proplnt, called "Force Constant", is defined by

$$F = nRT_o$$

This force can be determined experimentally by burning a chge of proplnt in a closed chamber and measuring the max pressure produced and using Abel's equation (above), along with suitable cooling corrections. This requires a knowledge of η which can be detd simultaneously by firing a series of chges of different masses and measuring the corresponding pressures

The table includes values of the force for a number of std & experimental gun proplnts. Force & other thermodynamic parameters of proplnts can be calculated theoretically if the necessary thermochemical data are available. Results of such calculations are given in Ref 2

Refs: 1) SACMS, Ballistics (1951), pp 17, 22 & 53 2) P.G. Baer & K.R. Bryson, "Tables of Computed Thermodynamic Properties of Military Gun Propellants", BRL Memo Rept **1338** (1961) 3) Staff, "Interior Ballistics of Guns", AMCP **706–150** (1965), pp 1–15

Table F8a

Calculated Thermochemical Values for US Propellants

Propellant	Use	Specification Number	Isochoric Flame Temp, °K	Gas Vol moles/g	Force ft lbs/lb x 10^{-3}
IMR	Small Arms	JAN-P-733			
M1	Artillery	JAN-P-309	2417	0.0453	305
M2	Artillery	JAN-P-323	3319	0.0390	360
M5	Artillery	JAN-P-323	3245	0.0394	355
M6	Artillery	JAN-P-309	2570	0.0443	317
M7	Mortar	JAN-P-659	3734	0.0354	368
M8	Mortar	JAN-P-381	3695	0.0371	382
M9	Mortar	JAN-P-20306	3799	0.0362	382
M10	Recoilless	PA-PD-123	3000	0.0407	339
M12	Small Arms	JAN-P-528	2996	0.0404	336
M14	Artillery	JAN-P-309	2710	0.0434	327
M15	Artillery	PA-PD-26	2594	0.0465	336
M17	Artillery	PA-PD-26	3017	0.0434	364
M18	Small Arms	FA-PD-26A	2577	0.0422	319
T18	Recoilless	PA-PD-329	2938	0.0446	346
T20	Artillery	–	2388	0.0479	314
T25	Recoilless	PA-PD-329	3071	0.0413	353
M26	Recoilless	PA-PD-329	3081	0.0416	356

Force de l'explosif (Force spécifique, ou Force théorique) de Sarau, also called Force, Pression spécifique (Fr for Force of an Explosive). This is an arbitrary value proposed by Sarrau for the estimation of the performance of explosives. The value may be calculated from the equation given in Marshall 2(1917), p 467, as follows:

$$f = pv = \frac{v_o Q}{C} \cdot \frac{p_o}{273} + \frac{p_o v_o T_1}{273}$$

where:

f = "force" expressed in kg/sq cm exerted by the expln of 1 kg in 1 liter

p = pressure of expln expressed in atmospheres

v = volume of the vessel in which the expln takes place

Q = quantity of heat set free by the expl of unit weight

v_o = "specific volume" of the gaseous products from a unit weight of expl calculated to 0°C and standard pressure, p_o

C = sum of the mean specific heats of the products at the temp of expln. It is a very uncertain quantity

T_1 = the absolute temperature of the expl before it is fired

Pepin Lehalleur (1935), p 41, gives the following simplified formula:

$$f = \frac{p_o V_o T}{273.1}$$

where:
p_o = atmospheric pressure equal to 1.0333kg/sq cm
V_o = volume of gases developed on expln in liters per kg & calculated at 0° and 760mm, with water as vapor
T = absolute temperature developed on expln

The following values for f are given by Pepin Lehalleur(1935), pp 49, 54 & 78: TNT 8910 & 8080; PA 9850 & 8950; 92/8 Blasting Gelating 12300; PETN 12700; RDX 12619 & 12600; Tetryl 10800; NC(13% N) 10730; MF 5630; LA 4339; NG 7450; Nitrogen Sulfide 6479; Black Powder 2810; NH_4NO_4 5575; NH_4ClO_4 4860 in kg/sq cm

The force (f) was used by Kast in his formula for the theoretical detn of brisance (B), called "pouvoir brisant" in French:

$$B = fdD$$

where:
d = density of an expl and
D = velocity of detn
[See also Vennin, Burlot & Lécorché(1932), p 53 and H. Muraour, MAF **22**, 133–70(1948), La force et le covolume des poudres colloidales.

In the paper of R.H. Kent, JApplPhys **13**, 348–54(1942), the following definition of Force was given on p 349

Force of an explosive is roughly an index of the weight lifting or heaving ability. Suppose the explosion takes place in a large closed chamber with rigid walls. If v is the volume of the chamber and p is the pressure produced by explosive gases as they fill the chamber, the "force" can be defined as the product of v and p per unit wt of expl. If the chamber volume is sufficiently large to permit the perfect gas law to hold (approximately), the product pv is practically independent of the volume for a given expl. If an expl detonates in a confined space, this definition is no longer valid, since the density is so high that the perfect gas law is no longer a good approximation. For such cases, one takes the force as proportional to the product of the number of the gram-molecules per gram of expl and the temperature. It is evident that if the density is small, this definition of force is equivalent to the one given above

The forces of a number of expls were detd experimentally when they deflagrated or burned, but no reliable direct measurements of forces produced on detonation have been obtd. However, it is possible to calculate the forces from thermochemical data. Some of these data were obtd by an analysis of the band spectra of the various molecules concerned by quantum-mechanical methods, which permitted one to calculate the specific heats and equilibrium constants as functions of the temperature. Others obtd by direct measurements of the heats of formation of the various substances from their elements. The results obtd by these methods for deflagrating and burning expls agreed fairly closely with the forces as obtd by direct measurements

In making the above mentioned calculations for explosives, it is convenient to assume some pressure in the explosion since theory and experiment both indicate that the compn of the products of expln depends upon the pressure. For example, a very high pressure will tend to produce solid carbon or smoke. It is for this reason that a cloud of black smoke produced on expln of a charge of TNT indicates that high pressure occurred and that, therefore, the charge detonated and did not merely deflagrate or burn (which would produce a relatively low pressure)

The following forces, in inch lb/lb calcd at 1500000psi (100000 atm), listed by Kent are shown here in Table F9: TNT 3627000, Tetryl 4728000, Picric Acid 3999000 and Blasting Gelatin 4974000

It was mentioned that on deton of TNT

Table F9

Percentage Products (by Weight) of Explosion (or Detonation) for the Following Explosives

Explosive	CO_2	CO	H_2O	H_2	N_2	OH	NO	O_2	O	C	CH_4	NH_3	HCN
TNT	7.3	59.7	3.2	0.7	16.2	–	–	–	–	5.7	3.5	1.2	2.5
Tetryl	8.4	59.8	4.8	0.8	22.3	–	–	–	–	–	0.5	1.2	2.2
Picric Acid	12.5	63.1	4.3	0.5	17.0	–	–	–	–	–	0.5	0.7	1.4
Blasting Gelatin	49.4	7.5	18.5	0.1	16.8	2.2	2.7	2.7	0.1	–	–	–	–

there was produced a large amt of smoke. Actually, the gas produced on expln of TNT contd 5.7% carbon by weight, as can be seen from Table F9 giving values calcd by Kent using equilibrium constants given by B. Lewis & G. von Elbe in JACS **57**, 612(1935)

Since the term *pressure* (p) developed on deton or expln is in the formula for calculating the force, we are including its description as given in the paper of Kent, p 352. Due to the fact that such pressures are enormous, no experimental procedure for direct measuring them is known. Although these pressures have not been directly measured, there is a certain phenomenon accompanying a detonation which enables one to assign at least an approx lower limit of the pressure developed

It is known that at low pressure and high temperature all the carbon would unite with oxygen and hydrogen and there would be no smoke. In this case the total volume (v) of gases will be greater than if the smoke were produced, which happens at high pressures. From the knowledge of thermochemical data, one can compute approx the pressure at which the smoke begins to form for various expls or proplnts. For example, it has been calcd that the smoke forming pressure for NC is 1100000psi

Another important characteristic of an explosive, which might influence the force is the *density*, abbrd as d. This is especially important in underwater explosions, such as of sea mines, torpedoes, depth bombs and bombs dropped alongside a ship. If the volume in which the expln occurs is large, compared with the vol of the expl itself, the free space available for the motion of the molecules of the expl gas, is large compared with the molecules themselves. Under these conditions, the perfect gas law holds approximately, that is the product of pressure and volume (pv) is approx constant and is approx independent of v. It follows that under these conditions with a given amt of explosive, the pressure is approx proportional to 1/v

Consider now an explosion taking place in a confined space, such as that of a bomb under water. Under these conditions, the volume occupied by the gaseous molecules is no longer small compared with the total vol available and the perfect gas law no longer holds. However, the previous statement holds if a new definition of v is given, namely if it is taken not as total volume, but as the "free volume" available for the motion of the molecules. Since the space taken up by the molecules is approx proportional to their number and since the number for ordinary explosives is approx proportional to their weight, it follows that the space occupied by the molecules themselves is approx proportional to the wt of the explosive

Consider further that two bombs of the same size are exploding under water. The bombs are filled with two different explosives, having the same force as measured in inch lb/lb, but having different densities. Assume that the expl of No 2 has twice the density of that of bomb No 1 and thus its weight will be twice as great. Since the force per pound is the same, the total force of bomb No 2 will be twice as great as that of bomb No 1. In addition, the denser explosive will, in general, have more molecules than the other and these will occupy more space. Since the pressure varies inversely as the free space (which is the total space minus the space effectively occupied by the molecules) the pressure produced by the denser explosive should be considerably higher than twice that produced by the less dense expl

In certain applications, especially in submarine work, the magnitude of the pressure obtainable should be of great importance, hence the desirability of high density

Following are maximum loading densities for some expls: NG 1.60, Blasting Gelatin 1.63, TNT 1.55 (average d cast in shell), Tetryl 1.65 & PA 1.69(Kent, p 351)

Another important characteristic of an explosive which might influence its force is the *energy*, which is defined as the heat (in calories per unit weight) produced on detonation or explosion. It may be either calculated or determined experimentally using a small, thick-walled calorimetric bomb. When a charge of an expl first detonates the gases produced are at very high pressure,

but they are moving at a relatively low velocity. As the gases immediately begin to expand, the heat energy is converted in part into kinetic energy of motion of the gas, while another part is used in pushing aside the atmosphere. At a short distance from the point of expln, the pressure in the expanding gases is very much reduced, but the envelope has attained a velocity of the order of 10000 to 15000 ft/sec. A gas of high density moving at very high velocity is very destructive

The following values for energy in cal/g are given by Kent (p 352): TNT 782, PA 830, Tetryl 988 and Blasting Gelatin 1364

When a bomb is dropped inside a building, the most important characteristic of the expln from a military point of view is probably the force. For a bomb of a given weight, the outward thrust on the walls depends mainly on the force. If on the other hand the bomb is dropped in the open alongside a ship or a building, the effect produced would depend not only on the force but also on the rapidity with which the explosion takes place and this is detd by its *rate of detonation*, abbreviated as D (Kent, p 350)

This destructive effect depends also on the ability of an expl to produce damaging *blast effect*, which is caused mainly by exploding gases which attained high velocities in the vicinity of the point of expln. Since this requires heat of high temperatures, it is a plausible assumption that blast effect is proportional to the energy. On this basis the relative blast effect of several expls, with TNT taken as unity, was given by Kent on p 352: TNT 1.00, PA 1.06, Tetryl 1.26 and Blasting Gelatin 1.74

It must be noted that for closely confined explns, the force (f), the rate of deton (D) and density (d) are all of great importance. In order to obtain a means for rating confined expls, all these factors must be taken into consideration. This was done by Kast, who proposed calling the product of these values *brisance* or *shattering effect* and designated it as B (See the beginning of this item and in Vol 2 of Encycl, p B265-R)

The comparative brisance by Kast, B = fdD, as given by Kent on p 352 is: TNT 1.00, PA 1.23, Tetryl 1.47 & Blasting Gelatin 1.59

Force or Impetus. See Vol 4, p D391-R under Detonation (and Explosion) Impetus and Available Energy

Force de pénétration. Fr for Penetrating or Perforating Power

Forcement dans les bouches à feu. Fr for Forcing in Gun Barrels, which will be described under Résistances passives et forcement dans les bouches à feu (Fr for Passive Resistances and Forcing in Gun Barrels)

Force propulsive. Fr for Propelling Force

Force spécifique. Fr for Specific Force

Forcite américaine. See under FORCITES

Forcite antigrisouteuse. See under FORCITES

Forcite extra. See under FORCITES

Forcite Gelatine. See under FORCITES

Forcite de Lewin. See under FORCITES

Forcite supérieure. See under FORCITES

FORCITES. Accdg to Daniel (Ref 1) and Giua (Ref 9), Forcites are Gelatin Dynamites patented in 1880 by Swedish Officer Lewin.

Gody (Ref 2, p 374) describes under the title *Forcite de Lewin or Forcite supérieure* the compns contg NG 65–75, "cellulose acétique" 4, dextrine 4 & poudre de mine (BkPdr) 30–20%. "Cellulose acétique" was prepd by treating cotton linters with a mixture of Na acetate and sulfuric acid, followed by washing with a large quantity of water, pulping to the size of fibers used in the manuf of paper, and drying. The resulting acetate was partly sol in NG forming a liquid jelly. Lewin also prepd *Forcite n° 1* which contd 48% NG, *Forcite n° 2* with 30% NG and *Forcite n° 3* with 20–25% NG (Ref 2, p 374). Under the title of *Forcite américaine*, Gody (Ref 2, p 375) gave the following: NG 7.9–70, NC 0.1–4.0 & absorbent 92–20%. Compn of the absorbent was $NaNO_3$ 84.1, sulfur 9.5, colophony 3.7 & wood tar 2.7%. Accdg to Thorpe (Ref 4 as quoted in Ref 7), Forcites are plastic expls invented in the USA in 1881, and that one of the best compns contained NG 75, CC (Collodion Cotton) 7 & K nitrate 18%. Dextrine or pulped cotton could replace part of the CC. Another Amer Forcite was known as Forcite Gelatin. It consisted of NG 95–96 & soluble NC 5–4% (Ref 8, p 36). Forcites were manufd in Belgium at the Baelen-sur-Nèthe, and Gody gives on p 374 eight compns manufd there. Marshall (Ref 3), Naoúm (Ref 5) and Davis (Ref 6) list only seven of the Gody's eight compns (See the table), omitting another *Extra* which contained NG 74, NC 6, dried woodflour 5.5, KNO_3 14 & $MgCO_3$ 0.5%

Table F10

Belgian Forcites

Components	Extra	Supérieure	Supérieure F	No 1	No 1P	No 2	No 2P
NG	64	64	64	49	49	36	36
NC	3.5	3	3	2	2	3	2
$NaNO_3$	–	24	–	36	–	35	–
KNO_3	–	–	23	–	37	–	46
AN	25	–	–	–	–	–	–
Woodmeal	6.5	8	9	13	11	11	–
Bran	–	–	–	–	–	14	15
$MgCO_3$	1	1	1	(added) 1	1	1	1

Note: Forcite Extra is actually an Ammonia Gelatin Dynamite. The same Forcites as above are listed by Giua (Ref 9) as French Forcites. Gody also lists on pp 702, 706 & 715 the following *Forcites antigrisouteuses* (See Table F11)

Table F11
Forcites antigrisouteuses

Components	Designations		
	nº 1[a] de Baelen-sur-Nèthe	nº 2	nº 3[a]
AN	70	–	–
NG	29.4	44	26
CC	1	–	–
$MgSO_4$	–	44	–
Cellulose	–	12	–
KNO_3	–	–	34
$Ba(NO_3)_2$	–	–	1
Wheat flour	–	–	38.5
Bark flour	–	–	1
Na_2CO_3	–	–	0.5

a – Compn of nº 1 adds to 100.4%, while that of nº 3 adds to 101%

Refs: 1) Daniel (1902), 302–304 2) Gody (1907), 374, 375, 702, 706 & 715 3) Marshall 1(1917), 372 4) Thorpe 2 (1917), 6 5) Naoúm NG(1928), 329 (Table) 6) Davis (1943), 346 7) Clift & Fedoroff 2(1943), pp F2 & F3 8) Fedoroff & Clift 4 (1946), 36 9) Giua, Trattato VI(1)(1959), 344(Table)

Fördit (Ger). Permissible Gelatin Dynamites exist in several formulations, such as shown in Table F12

Table F12

Component	Designation		
	Fördit	Fördit I	Fördit IV
AN	41.0	37.0	38.0
NG	23.0	25.5	21.0
CC	1.0	1.5	1.0
MNT	3.5	5.0	5.0
Glycerin	8.7	3.0	3.0
Cereal or potato flour	–	–	12.0
KCl	22.0	24.0	19.0
China clay	0.1	–	–
Amm oxalate	–	–	1.0
Dextrine	0.7	4.0	–

Refs: 1) Naoúm, NG(1928), 407 & 411 2) Clift & Fedoroff 2(1943), p F3 3) Fedoroff et al, PATR 2510 (1958), p Ger 52-L

Foreign Ammunition and Weapons. See under specific ammunition or weapon item, eg, bombs or specific country, eg Belgian, British

Foreign Artillery Fuzes of WWII. See Vol 4, pp D923 to D925

Foreign Bomb Fuzes of WWII. See Vol 4, p D1008 to D1010

Foreign Cannon Propellants. See Vol 2, p C37-L

Foreign Commercial (Industrial) Explosives. See Vol 3, pp C437 to C444 (For Non-permissible) and pp C444 to C459 (For Permissible)

Forensic Ballistics are described by Sir Gerald Burrard, "The Identification of Firearms and Forensic Ballistics", H. Jenkins, London (1956)

Forensic Chemistry (Chemical Criminalistics). This branch of science relates to the application of chemical knowledge and techniques to the study of physical evidence connected with crime. Investigation of firearms, ammunition and expls is one of the duties of a forensic chemist

Refs: 1) A. Lucas, "Forensic Chemistry and Scientific Criminal Investigation", Longmans, Green, London (1935), Chap 7, Explosives; Chap 11, Fireworks 2) H. Söderman & J.J. O'Connell, "Modern Criminal Investigation", Funk & Wagnalls, NY (1945) 3) R.F. Turner, "Forensic Science and Laboratory Technics", C.C. Turner,

Springfield, Ill (1945) 4) Kirk & Othmer, Vol 4(1951), pp 848–57, "Forensic Chemistry" by P.L. Kirk (27 refs) 5) Ibid, 2nd Edn, not found

Forensic Science. It is discussed under the title "Forensic Emphasis Shifts to Broader Role" in C&EN, Feb 5, 1973, pp 13–15. It is stated that crime labs focus on development of techniques useful in high-incidence crimes, such as burglary and street assault. A problem of increasing frequency is detecting expls & other materials before they are used

Forestry Uses of Explosives. See Item j in Vol 1 of Encycl, p A114-L, under Agriculture and Forestry Uses of Explosives

Form and Measurement of Foreign Propellants. British, German, Italian, Russian and Japanese proplnts examined at Picatinny Arsenal during WWII included squares, perforated discs, single-perforated tubes, single perforated grains, multi-perforated grains, spheres and strips. The basic idea behind the use of the varied forms is connected with the desire for a proplnt with a high initial burning surface which these proplnts afford. The long tubular proplnts were used for field guns and fixed defenses, the short tubular proplnts with thin walls and the disc proplnts – for howitzers and mortars and the coated flake proplnts – for small arms. It has been usually considered that high NG, high nitrogen NC proplnts are used for mortars and howitzers, but a number of exceptions to this generalization have been found

Ref: A.J Phillips, PATR **1456**(1944)

Formaldehyde *(Monomeric Formaldehyde, Methanol, Formic Aldehyde, Formalin or Formol)*, HCH:O or CH_2O; mw 30.03, d 0.815 at −20°, vapor d 1.067 (air=1.0); fr p −118° (Walker and Faith et al); bp −19° (Walker and Faith et al); ignition temp 572°F; explosive limits (% by volume of air), lower 7, upper 73

Colorless gas with suffocating, pungent odor. The product known in commerce is its 37% aqueous solution. It is poisonous. Formaldehyde was first prepd in 1859 by A.M. Butlerov by hydrolyzing methylene acetate previously obtained by the reaction of methylene iodide with silver acetate. A.F. Hofmann, in 1868, prepd formaldehyde by passing a mixture of methanol vapors and air over a heated platinum spiral, serving as a catalyst. The following reaction takes place: $CH_3OH + \frac{1}{2}O_2$ (air) → $CH_2O + H_2O$. The present method of preparation is essentially the same as used by Hofmann, except that cheaper catalysts than platinum are used, such as Ag and Cu in multilayer gauzes or screens, vanadium and molybdenum oxides, iron oxides or other metal oxides. Limited amounts of formaldehyde are also prepd by the oxidation of natural gas and the lower petroleum hydrocarbons

All of these methods are described by Walker (Ref 2) and Faith, Keyes & Clark (Ref 3)

Procedure. Briefly the present method of manufacture by the methanol process is as follows:

Air is drawn thru methanol at such a rate that a mixture contg 30–50% methanol is obtd. The alcohol-air mixture passes thru a preheater (100–300°) in order to eliminate any alcohol in liquid form, and then goes to the reactor, where it remains only 0.01 second. The reactor contains either Ag or Cu multilayer gauze or screen, maintained at 450–600°. The resulting gaseous product is absorbed in water to give the commercial product

When metallic oxides are used as catalysts, a large excess of air is required – 5 to 10 times that required by the metallic catalysts

Although other methods of preparation, involving the hydrogenation of carbon oxides, the pyrolytic decomposition of formates etc, have been patented, they do not appear to have achieved commercial importance

Formaldehyde is sol in water and its 37% solution is the usual commercial product;

also sol in alc and eth

Both liquid and gaseous formaldehyde polymerize readily at low temperatures and can be kept in the pure monomeric state only for a limited time. Because of these facts, formaldehyde is sold and transferred either in solution or in polymerized form, such as *paraformaldehyde* and *trioxane*, described here under Formaldehyde Polymers

Commercial, 37% solution of formaldehyde (So-called Methanol-free)

Clear, colorless liquid, d 1.1119 at 25°/25°; bp 101°; pH about 3.0; flash point 185°F; ignition temp about 806°F; methanol content about 0.4% by wt; RI 1.3777 at 18°

Uses of Formaldehyde. Consumption in the US increased from about 8.5 million pounds in 1914 to 500 million pounds in 1943

As a gas, it is used as a disinfectant. As aqueous solution, it is used as a parent compound for preparation of numerous chemicals used in industry, such as in the manufacture of explosives, plastics, resins (eg phenol-formaldehyde resins) etc (See further under Formaldehyde and Derivatives, Explosives obtained therefrom)

When reacted with glycerin, it forms "glycerol formals" (qv)

Qualitative and quantitative determinations of formaldehyde are given in Ref 2 (1944), pp 244–75 and in 3rd edn, pp 467–510

Refs: 1) Beil 1, 558, (280) & [615] 2) J.F. Walker, "Formaldehyde", Reinhold, NY (1944) & 1964 (3rd ed) 3) W.L. Faith, D.B. Keyes & R.L. Clark, "Industrial Chemicals", Wiley, NY (1950), pp 338–344 4) CondChemDict (1961), pp 510–11 & (1971), p 391 5) Kirk & Othmer, Vol 10 (1966), pp 77–98 6) ASTM Method D2378 (1968) for Formaldehyde Reagent 7) Sax (1971), 782

Formaldehyde Polymers. Polymer formation is one of the most characteristic properties of the formaldehyde molecule. Two types of polymers exist: $.CH_2.O.CH_2.O.CH_2.O.$ and $.CH(OH).CH(OH).CH(OH).C.$, but only one, the first type, is called "formaldehyde polymer". Other name is "polyoxymethylene". Representatives of the 2nd type are encountered in the "polyhydroxyaldehydes". Carothers divides the polyoxymethylenes into two groups: A- and C-polymers. A-polymers are formed by the polymerization of anhydrous monomeric formaldehyde:

$$H_2C{:}O + H_2C{:}O + H_2C{:}O + \ldots \rightarrow .CH_2.O.CH_2.O.CH_2.O.,$$

while the C-group is derived from aqueous formaldehyde involving formaldehyde hydrate (methylene glycol):

$$HO.CH_2.OH + HO.CH_2.OH + HO.CH_2.OH + \ldots \rightarrow HO.CH_2.O.CH_2O \ldots CH_2OH + (n-1)H_2O.$$

These compounds are *polyoxymethylene blycols.* Walker (Ref 1, p 65 & Ref 3, p 142) gives a table describing the properties of various formaldehyde polymers

All of these polymers are linear with the exception of *sym-trioxane* [*α-trioxymethylene*) $(HCHO)_3$] and *tetraoxymethylene* $(HCHO)_4$, both of which are true polyoxymethylenes, although they are not prepd by the polymerization of monomeric formaldehyde

Of all these polymers, **Paraformaldehyde** is the only one which is manufd on a large commercial scale. It is also known as *Paraform, Polyformaldehyde* or **Polyoxymethylene.** It can be represented by formulas $(HCHO)_n$ or $HO(CH_2O)_nH$ in which n is equal to 6. It is wh solid with sl odor of formaldehyde; insol in alc, eth or w; sol in strong alkali solns. Can be prepd by evaporating an aq soln of formaldehyde. Causes irritation of skin, eyes, nose and throat. Used as disinfectant, fungicide and in prepn of some resins, artificial horns, ivory and explosives

The role of paraformaldehyde in prepn of HMX was discussed by Castorina of Picatinny Arsenal (Ref 2)

The polymer, **sym-Trioxane,** also known as *α-Trioxymethylene, Metaformaldehyde, Triformol* or *Trioxin,* can be represented as $(HCHO)_3$ or $\underline{CH_2OCH_2OCH_2O}$, consists of wh crysts with formaldehyde odor, mp 62°, bp 115°, fl p (open cup) 113°F; sl sol in w; sol in alc & eth. Can be prepd by distilla-

tion of formaldehyde. Used as a nonluminous, odorless fuel, as disinfectant and in org synthesis

The polymer, **Tetraoxymethylene,** $(HCHO)_4$, or Tetroxane, $CH_2OCH_2OCH_2OCH_2O$, long, ndl like crysts, mp 112°; sol in acet, w, dil alkali & dil acid; vapor is stable at 200°; was obtd by heating a water-insol, polyoxymethylene diacetate of high mw. It is readily converted to a polyoxymethylene when in the solid state (Ref 1, 3rd edn, p 194)

Refs: 1) Walker (1944), pp 64–101 and in 1964 Edn, pp 140–205 2) T.C. Castorina, PATR **2481** (1958) (Role of paraformaldehyde in prepn of HMX) 3) CondChemDict (1961), p 848-R (Paraformaldehyde); p 1173-R (sym-Trioxane) & in 1971 Edn, p 657-R (Paraformaldehyde); p 899-R (sym-Trioxane) 4) Kirk & Othmer, Vol **10**, (1966), pp 81–86 by J.F. Walker

Formaldehyde and Derivatives (Explosives Obtained Therefrom)

A number of expls have been prepd from raw materials involving formaldehyde. Although most of the formaldehyde expls are prepd from formaldehyde derivatives, several expls can be prepd directly in one operation from formaldehyde or anhydrous formaldehyde, or its polymer, *paraformaldehyde*. Walker (Ref 38), in his book on formaldehyde, gives a brief description of expls derived from it. For convenience, we divide them into two groups – those obtained directly from formaldehyde and those from formaldehyde derivatives

1. **Explosives Obtained Directly from Formaldehyde:**

a) When concd nitric acid is mixed with formaldehyde, no visible reaction takes place, but if the mass is stirred, a violent, nearly expl reaction takes place (Ref 5)

b) When formaldehyde soln is nitrated at about 5°, the liquid expl, *Methyleneglycol Dinitrate* is obtained (Ref 25)

c) When anhydrous CH_2O, dissolved in ether, is treated with anhydrous H_2O_2, the liquid expl, *Methylol Hydrogen Peroxide*, is obtained (Ref 29). From the same reagents, a solid expl, *Dimethylol Peroxide* (qv) was also obtd (Refs 3 & 10). The methylol peroxide derivatives may be converted to peroxides of trioxane and tetraoxymethylene, such as liquid *Pertrioxane* and solid *Tetraoxymethylene Diperoxide*, both of which are very expl (Ref 21)

d) Formaldehyde peroxides are also formed by the reaction of peroxide on paraformaldehyde

3) When formaldehyde (especially the commercial grade, ie containing paraformaldehyde) is treated with sodium peroxide, the reaction proceeds with expl violence (Ref 2)

f) When formaldehyde is treated with 3% H_2O_2 in the presence of $(NH_4)_2SO_4$, an expl product, *Hexamethylenetriperoxidediamine*, (Ref 1) is formed. The same product was also obtd from hexamethylenetetramine and H_2O_2 (Refs 7 & 14)

g) When formaldehyde is treated at 40–50° with H_2O_2 in the presence of hydrazine sulfate, an expl peroxygen compound called *Trimethyleneperoxide Azine* is obtd (Ref 7, p 490)

h) When formaldehyde (or paraformaldehyde) is treated with gaseous HCl and the resulting dichlormethyl ether, $Cl.CH_2.O.CH_2.Cl$, is nitrated, an expl oil, *Nitromethoxymethyl Nitrate*, is obtd (Ref 6)

i) When formaldehyde is treated with hydrogen peroxide in presence of ethylamine, a liquid expl, *Dimethyleneperoxideethylamine*, is obtd (Ref 7, p 497)

j) When formaldehyde reacts with hydrogen peroxide and urea, an expl, *Tetramethylenediperoxidedicarbamide*, is obtd (Ref 38, p 332 & Ref 4)

k) Dr Hans Walter prepd during WWII the explosive *Formit* (qv) by heating under reflux a mixture of 30% commercial formaldehyde with Amm Nitrate

2. **Explosives Obtained from Derivatives of Formaldehyde:**

An important group of expl organic nitrates is produced by the action of nitric acid on polyhydroxy compounds formed by the reaction of formaldehyde with ether al-

dehydes, as well as with ketones and nitroparaffins

a) When formaldehyde is treated with acetaldehyde and the resulting compd (pentaerythritol, or PE) is nitrated, the final product is *Pentaerythritoltetranitrate*, one of the most powerful expls, being only slightly less so than RDX (Ref 38, p 330)

b) The following expls, derived from PE, were patented in Germany (Ref 28): Pentaerythritol Chlorohydrin Nitrates, PE Diacetate Dinitrate, PE Diformate Dinitrate, PE Dimethyl Ether Dinitrate, PE Trinitrate Mononitrobenzoate, PE Diglycollic Ester Tetranitrate, PE Monomethyl Ether Trinitrate. In addition to these, Wyler (Ref 29) patented the expl PE Tetraacetate Tetranitrate

c) Due to the fact that crude PE always contains variable amounts of by-product dipentaerythritol, after the nitration of PE there is always present some *Dipentaerythritol Hexanitrate* (qv), which is a solid expl (Ref 30)

d) When formaldehyde is reacted with propionaldehyde and the resulting pentablycerol, $CH_3C(CH_2OH)_3$, is nitrated, a liquid expl, *Trimethylolmethylmethane Trinitrate*, is obtd (Refs 15 & 22)

e) When formaldehyde reacts with isobutyraldehyde and the resulting pentaglycol is nitrated, an expl *Pentaglycol Dinitrate* is obtd (Ref 18)

f) When formaldehyde reacts with n-butyraldehyde and the resulting trimethylolmethane is nitrated, a solid expl Dinitrate is obtd (Ref 17)

g) Formaldehyde reacts with ketones yielding products which give expls on nitration, such as *Anhydroenneaheptitol Pentanitrate* (Ref 37)

Friederich (Ref 23) prepd expl nitrates from products of reaction between cyclopentanone and cyclohexanone

h) When formaldehyde reacts with nitroparaffins in presence of alkalies, the following general reactions take place: $R.CH_2.NO_2 + 2CH_2O(aq) \rightarrow R.C(CH_2OH)_2.NO_2$. These reactions were first studied by L. Henry in 1895

Nitration of these compds yields products of the formula $R.C(CH_2.O.NO_2)_2.NO_2$, some of which are useful as expls. For example, *Nitroisobutylglycerin Trinitrate* (Ref 1, p 331), *Dimethylolmethylnitromethane Dinitrate* (Refs 17, 35 & 39) and *2-Nitro-isobutanol Nitrate* (Ref 35)

i) When formaldehyde reacts with ammonia, the following reaction takes place: $6CH_2O + 4NH_3 \rightarrow (CH_2)_6N_4 + 6H_2O$. The resulting product, *Hexamethylenetetramine*, is a parent substance for a number of expls, the most important of which is RDX or Cyclonite. Described in Vol 3 of Encycl, p C611ff as CYCLOTRIMETHYLENETRINITRAMINE

j) Other expls derived from hexamethylenetetramine are: *Hexamethylenetriperoxide Diamine* or HMTD (Ref 38), p 289 & 331 and Refs 12, 24 & 30); *Hexamethylenetetramine Perchlorate* (Ref 38, p 331 and Ref 34); *Hexamethylenetetramine Trinitro-m-cresylate* (Ref 38, p 331 and Ref 9); *Cyclotrimethylenetrinitrosamine* (Ref 38, p 332 and Ref 34)

Refs: 1) A. Bayer & V. Villiger, Ber **33**, 2486 (1900) 2) L. Vanino, ZAnalChem **41**, 619–20 (1902) 3) H.J.H. Fenton, JCS **106**(I), 1121 (1914) 4) G. von Girsewald & Siegens, Ber **47**, 2466 (1914) 5) M. Verwern, ArchivGesamtePhysiol **167**, 289–308 (1917) 6) A. Mereschi, AttiRAccadLincei (Roma) (5), **28**(I), 277–80 (1919) & JCS **116**(I), 385 (1919) 7) F.C. von Girsewald & H. Siegens, Ber **54**, 490–91 & 492–97 (1921) 8) E.R. von Herz, GerP 286527 (1922) 9) R.L. Datta et al, JACS **45**, 2430 (1923) 10) H. Wieland & A. Wingler, Ann **431**, 301–22 (1927) 11) M. Bamberger & J. Nussbaum, SS **22**, 125–28 (1927) 12) G.A. Taylor & W.H. Rinkenbach, JFranklInst **204**, 374 (1927) 13) F.H. Bergeim, USP 1691955 (1928) 14) G. Marotta & M.E. Allessandrini, GazzChimItal **59**, 942–46 (1929) 15) E.R. von Herz, GerP 474173 (1929) 16) W. Friederich & W. Brün, Ber **63**, 2881–90 (1930) 17) C.P. Spaeth, USP 1883044 (1932) 18) C.P. Spaeth, USP 1883045

(1932) 19) W. Friederich & W. Brün, SS **27**, 73–6 & 125–27 (1932) 20) Stettbacher (1933), 179 21) A. Rieche & R. Meister, Ber **66**, 718–27 (1933) 22) Bombrini Parodi-Delfino, FrP 771599 (1934) 23) W. Friederich, USP 1962065 (1934) 24) A. Schmidt, SS **29**, 263 (1934) 25) G. Travagli & A. Torboli, ItalP 333080 (1935) 26) A. Rieche & R. Meister, Ber **68**, 1465–73 (1935) 27) Nuevos Explosivos Industriales SA, FrP 783682 (1935) 28) Westfälisch-Anhaltische Sprengstoff AG, GerP's 638432 & 638433 (1936) 29) J.A. Wyler, USP 2086146 (1937) 30) M. Patry, SS **32**, 177 (1937) 31) O. Hassel, NorwegP 57831 (1937) 32) W.deC. Crater, USP 2112749 (1938) 33) J. Meissner, USP 2208934 (1940) 34) L. Bellini, AnnChim-Applicata **31**, 125–29 (1941) 35) B.M. Vanderbilt, USP 2241492 (1941) 36) C. Ellis, USP 2274629 (1942) 37) Davis (1943), 227 & 279 38) J.F. Walker, "Formaldehyde", Reinhold, NY (1944), 127–28, 134 & 330–32 39) R.A. Cooley, ChemInd **59**, 645 (1946) (Account of Axis process for RDX manuf during WWII) 40) R.D. Cadle & W. Moshier, USP 2871224 (1959) (Proplnt prepd by a combination of a formaldehyde-aniline condensation product with perchloric acid) 41) M. Scalera & M. Bender, USP 2826485 (1958) (A water-resistant expl compn contg AN & a solid fuel mixed with an ethylenic monomer contg methylolamide groups) 42) A.J. Courtier, FrP 1170356 (1958) (Stabilized AN against shock & temp by combination with a resin produced from formaldehyde & dicyandiamide or cyanamide) 43) A. Yamamoto, JapP 3074 (1959) (A proplnt consisting of a solid formaldehyde polymer & oxidizing agent such as a permanganate, nitrate or perchlorate) 44) J.F. Walker, "Formaldehyde", Reinhold, NY (1964), pp 288 (PETN), 528–30 (RDX & HMX), 532–33 (HMTD, Nitrogen Trichloride, etc) & 578–81 (Expls)

Formaldehyde Cyanohydrin. See Glycolonitrile

Formaldehyde Gelatin or **Glutofom.** See under Formaldehyde-Starch Mixture

Formaldehyde Peroxide. See Dimethylol Peroxide in Vol **5** of Encycl, p D1357

Formaldehyde-Starch Mixture is, accdg to Hackh's (1944), p 53-L, an antiseptic powder *amyloform*, used like iodoform. He calls (p 381-R) the product of reaction of formaldehyde & starch *glutol*, while the name of *glutol* or *glutoform* is given in CondChemDict (1961), p 540-L to product obtd by the action of formaldehyde & gelatin. This product exists as a clear transparent mass which may be pulverized or a white to yel powder. It is insol in cold w but sol in hot w, under pressure

Formalin. Same as Formaldehyde

Formamide and Derivatives

Formamide or *Methanamide*, $H.CONH_2$; mw 45.04, N 31.11%, mp 2.6°, bp 114°(18 torr), 103°(9 torr), sp gr 1.1339 at 20°, n_D 1.4481 at 20°, heat of combustion at const vol 136.7kcal/mole; sol in w & alc; insol in benz & chlf; sl sol in eth. Prepd by heating ammonium formate at 230° in a closed tube (Ref 2), by heating ethylformate with NH_3 (Ref 3) or heating ammonium formate with urea at 140° (Ref 4). Used as an industrial solvent

Refs: 1) Beil **2**, 26, (20), [36] & {54}
2) A.W. Hofmann, JCS **16**, 73 (19863)
3) M. Behrend, Ann **128**, 335 (1863)
4) A.W. Hofmann, Ber **15**, 980 (1882)
5) CondChemDict (1961), 511-L 6) Kirk & Othmer **10** (1966), pp 103–08 7) Sax (1968), 782-R

Formamide Azide or **Azidoformamide.** See Carbamyl Azide in Vol **2**, p C40-L, under Carbamic Acid and Derivatives

Formamide Hydrochloride, $CH_3NO+HCl$; mw 81.51; ndls, insol in eth; stable when cold but explodes at 15° yielding NH_4Cl & CO (Refs 1 & 2). For this reason its use is limited as there are few reactions that can be handled with so unstable a compd
Refs: 1) Beil **2**, {59} 2) P.L. Magill, IEC **26**, 613 (1934)

Formamide Perchlorate, $H.CO.NH_2.HClO_4$; mw 145.51. Accdg to VanDolah it combines low sensitivity to shock with high oxygen content
Refs: 1) Beil, not found 2) R.W. VanDolah, NavalOrdnMemorandumRept **10068** (1949) 3) CA, not found thru 1971

Formamidine. Accdg to Hackh's (1944), p 352-L *formamidine* is a compd contg the bivalent –N:CH.NH– radical, while *guanyl* (p 391-R) is the monovalent radical $-C(:NH).NH_2$. Since the compds described below are listed in Beil and other refs under Formamidine, we are following their nomenclature and giving the name of Guanyl as an alternate

FORMAMIDINES

Azidoformamidine or **Guanylazide** (Triazoformamidin, Carbamid-imid-azid or Kohlensäure-amidin-azid, in Ger), $N_3C(=NH)NH_2$; mw 85.07, N 82.33%, OB –65.9%. Prepd from the nitrate salt (which was prepd from aminoguanidine nitrate and nitrous acid) by treatment with NaOEt in alc, but quickly rearranged to the Tetrazole. Consequently, it is known only as its salts
Ref: 1) Beil **3**, 130, (60) & {239}

Azidoformamidine Hydrochloride, $N_3C(=NH)NH_2.HCl$; mw 121.54, N 57.63%, OB –46.1%; wh crysts, mp 126–46° (dec). Prepd by refluxing in concd HCl 1-Guanyl-4-(5-tetrazolyl)-tetrazene Nitrate (Ref 2)
Refs: 1) Beil, not found 2) S.H. Patinkin, J.P. Horwitz & E. Lieber, JACS **77**, 562–7 (1955) & CA **50**, 973h (1956)

Azidoformamidine Nitrate, $N_3C(=NH)NH_2.HNO_3$; (See also Vol **1**, p A638-L), mw 148.08, N 56.75%, OB –10.8%; plates or prisms (from alc), mp 129°, expl at higher temp; sol in w & alc; insol in eth, sensitive to friction, Trauzl Test value 400 cc, deton vel 5200 m/sec. Prepd from action of nitrous acid on aminoguanidine nitrate (Ref 2) or treating a very cold soln of cyanamide in hydrazoic acid with cooled concd nitric acid (Ref 5)
Refs: 1) Beil **3**, 130, (60) & {239} 2) J. Thiele, Ann **270**, 46 (1892) 3) K.A. Hofmann et al, Ber **43**, 1095 (1910) 4) W.S. McEwan & M.W. Rigg, JACS **73**, 4725–27 (1951) & CA **46**, 4350 (1952) (Heat of comb) 5) W. Sauermilch, Explosivst **9**, 256–57 (1961)

Azidoformamidine Perchlorate, $N_3C(=NH)NH_2.HClO_4.1.5H_2O$; mw 185.53 ($-H_2O$), N 37.75% ($-H_2O$), OB 0.0%; plates (from alc), sol in w. Prepd from aminoguanidine nitrate, 20% $HClO_4$, and sodium nitrite (Ref 2). Detonates under impact, burns like smokeless proplnt
Refs: 1) Beil **3**, (60) 2) K.A. Hofmann et al, Ber **43**, 1095 (1910) 3) ADL, PureExplCompds, Pt **4** (1952), p 567

Azidoformamidine Sulfate, $CH_3N_5+H_2SO_4$; mw183 ; colorless pltlts; readily sol in w; decomp in hot w and in dil sulfuric and nitric acids; deflagrates on heating
Refs: 1) Beil **3**, 130 & {239} 2) R. Hofsommer & M. Pestemer, ZElCh **53**, 386 (1949)

Azido-N-nitroformamidine, $N_3C(=NH)NHNO_2$; mw 130.07, N 64.61%, OB –12.3%; plates, mp 56° (dec, Ref 2), 79° (dec Refs 3 & 4); sol in eth, w, alc, chlf & acet; insol in benz. Prepd from Nitroaminoguanidine and nitrous acid at 0° (Refs 2 & 3). It is expl
Refs: 1) Beil, not found 2) T.E. O'Connor, G. Fleming & J. Reilly, JSocChemInd(Lond) **68**, 309–10 (1949) & CA **44**, 3443d (1950) 3) E. Lieber et al, JACS **73**, 1792–5, 2327–9

(1951) & CA **46**, 499b, 1987b (1952)
4) Ibid, JACS **74**, 2684–6 (1952) & CA **48**, 2693i (1954) 5) ADL, PureExplCompds, Pt **4** (1952), p 566

Azidoformamidine Picrate,
$N_3C(:NH)\cdot NH_2\cdot C_6H_2(OH)(NO_2)_3$; mw 314.18, N 35.66%, OB to CO_2 –51.0%
Refs: 1) Beil, not found 2) ADL, PureExplCompds, Pt **4** (1952), 564

Bromoformamidine Nitrate,
$BrC(:NH)NH_2\cdot HNO_3$; mw 185.98, OB –8.6%; colorless crysts, mp 65°, very unstable and expl, Trauzl Test value 170 cc vs 400 cc for the corresponding azido nitrate (qv). Prepd by slowly adding cooled nitric acid to a very cold soln of cyanamide in 40% HBr (Ref 2)
Refs: 1) Beil, not found 2) W. Sauermilch, Explosivst **9**, 256–57 (1961)

Bromoformamidine Perchlorate,
$BrC(=NH)NH_2\cdot HClO_4$; mw 223.43, OB 0.0%; colorless hygroscopic crysts, mp 132°, expl at higher temp, Trauzl Test value 120 cc vs 450 cc for the azido nitrate (qv). Prepd by slowly adding cooled concd $HClO_4$ to very cold cyanamide in 40% HBr (Ref 2)
Refs: 1) Beil, not found 2) W. Sauermilch, Explosivst **9**, 256–57 (1961)

Chloroformamidine Nitrate,
$ClC(=NH)NH_2\cdot HNO_3$; mw 141.52, OB –11.3%; colorless crysts, mp 120°, expl at higher temp; soly in w 7.9% at 0° and 22.2% at 30°. Prepd by treating cyanamide dihydrochloride in w with aq AN or from tech Ca cyanamide (Ref 2), also by adding cooled concd nitric acid to very cold cyanamide in HCl (Ref 3)
Refs: 1) Beil, not found 2) W. Sauermilch, Explosivst **9**, 74 (1961) 3) Ibid, 256–57

Chloroformamidine Perchlorate,
$ClC(=NH)NH_2\cdot HClO_4$; mw 179.00, OB 0.0%; cryst, mp 190°, expl at higher temp, soly in w 60.5% at 0° and 74% at 30°, Trauzl Test value 320 cc and detonvel 4950 m/sec vs 400 cc and 5200 m/sec for the azido nitrate (qv). Prepd by adding cooled concd $HClO_4$ to very cold cyanamide in HCl
Refs: 1) Beil, not found 2) W. Sauermilch, Explosivst **9**, 256–57 (1961)

1,1′-Azodiformamidine Dinitrate,
$(=NC(=NH)NH_2\cdot HNO_3)_2$; mw 240.14, N 46.66%, OB –13.3%; yel crysts, plates (from w), with rapid heating expl at 180–184°; sl sol in cold w; insol in alc & eth. Prepd by K permanganate oxidation of aminoguanidine nitrate in nitric acid (Ref 2)
Refs: 1) Beil, not found 2) J. Thiele, Ann **270**, 39 (1892)

1,1′-Azodiformamidine Dipicrate,
$[=NC(=NH)NH_2\cdot HOC_6H_2(NO_2)_3]_2$; mw 572.33, N 14.68% ($NO_2$), OB –55.9%; orn-red plates, mp 179–80° (dec). Prepd from the dinitrate (qv) by displacement (Ref 2)
Refs: 1) Beil, not found 2) J. Thiele, Ann **270**, 39 (1892)

1,1′-Azo-bis-nitroformamidine,
$[=NC(=NNO_2)NH_2]_2$; mw 204.11, N 54.90% OB –15.7%; red or orn-yel crysts (from CH_3NO_2), expl at 165°. Prepd from 1,1′-azodiformamidine dinitrate by conversion to *1,1′-Azo-bis-chloroformamidine*, $(=N(C=NCl)NH_2)_2$, and then treating with nitric acid (Ref 2)
Refs: 1) Beil, not found 2) G.F. Wright, CanJChem **30**, 62–70 (1952) & CA **47**, 3793 (1953)

4-Formamino-1,2,4-triazole,
HCONHNCH=NN=CH; mw 112.09, N 49.99%; crysts (from alc/eth), mp 117°, sol in cold w. Prepd by reacting dry formic acid and 4-amino-1,2,4-triazole at 100° (Ref 2)
Refs: 1) Beil **26**, 19 2) C. Bülow, Ber **42**, 2717 (1909)

Formates. See under FORMIC ACID AND DERIVATIVES

Formation, Heats of. See Vol 4, pp D369 & D370 and also in Tables A & B, pp D380 & D381

Formazans and Formazyls. According to Beil **16**, 4–5, *formazan* is the hypothetical parent compd HN=NCH=NNH$_2$, and *formazyl* is the radical HN=NC(–)=NNH$_2$; the latter may be considered the N-imino-N′-amino deriv of the guanyl or amidine radical HN$_2$C(–)=NH. This means that formazan may be called *formazylhydride* (Formazylwasserstoff, in Ger), although Hackh gives this name to the diphenyl deriv, PhN=NCH=NNHPh. Formazyl compds are said to have theoretically two isomeric forms: ArNHN=CRN=NAr′ and ArN=NCRNNHAr′ by M. Ragno & S. Bruno who prepd various nitro derivs [Gazz ChimItal **76**, 485–99 (1946) and CA **41**, 6542 (1947)]. For instance, the compd PhN=NC(CN)=NNHPh may be called N,N′-Diphenyl-C-cyanoformazan or N,N′-Diphenylformazylcyanide. Some of the formazan or formazyl derivs give expl salts while others form tetrazoles or other ring compds
Note: C-Methylformazan is called *Acetazidin* or Azidin in Beil **2**, 4, but the name azidin has been suggested for the radical N$_3$C(–)=NH, which is entirely different

N,N′-Diphenylformazan-C-Carboxylic Acid or **N, N′-Diphenylformazyl Formic Acid** (Formazylameisensäure in Ger), PhN=NC(CO$_2$H)=NNHPh; mw 268.28, N 20.89%; cherry-red ndls, mp 162–63° (dec); sol in chlf, benz & acet. Its *Silver salt*, AgC$_{14}$H$_{11}$N$_4$O$_2$, dark-brown crysts, expl on heating
Refs: 1) Beil **16**, 20, [8] 2) E. Bamberger & E. Wheelwright, Ber **25**, 3203 (1892) & JPrCh [2] **65**, 127 (1902)

N,N′-Diphenyl-C-Nitroformazan, PhN=NC(NO$_2$)N=NHPh; mw 269.26, N 26.01%; red ndls, mp ca 161° (dec, rapid heating); sol in alc, eth, benz & CS$_2$. Prepd from nitromethane, diazobenzene and a base
Refs: 1) Beil **16**, 25, [9] 2) E. Bamberger et al, Ber **27**, 157 (1894), **33**, 2059 (1900) & **34**, 578 & 586 (1901)

N, N′-Diphenylformazan-C-Azobenzene (Formylazobenzol or Benzolazoformazyl, in Ger), PhN=NC(N=NPh)=NNHPh; mw 328.38, N 25.60%; dark red leaflets, mp 162–3°; sl sol in hot alc; sol in acet, EtAc, chlf & benz. Some of its salts are expl, eg: *Copper (I) salt*, CuC$_{19}$H$_{15}$N$_6$, greenish-black powder and *Silver salt*, AgC$_{19}$H$_{15}$N$_6$, dark violet powder with metallic luster. Both salts explode on heating
Refs: 1) Beil **16**, 25–26 2) E. Bamberger et al, Ber **27**, 149 (1894) & JPrCh [2] **64**, 201 (1901)

N, N′-Diphenylformazylglyoxylic Acid (Formazylglyoxylsäure in Ger), PhN=NC(COCO$_2$H)=NNHPh; mw 296.29, N 18.91%. Exists in two stereoisomeric forms: the red, mp 166°, gives expl *Copper* and *Silver salts;* the yellow gives a *Silver salt*
Refs: 1) Beil **16**, 30 2) E. Bamberger et al, Ber **27**, 152 (1894) & JPrCh [2] **64**, 208 (1901)

Form Function. Consider that a chge of proplnt consists of a number of geometrically similar proplnt grains. Defining the size D of the proplnt grain as the least thickness to be burnt thru for complete combustion of the grain, denoting f as the fraction of D remaining at time t, z as the fraction of wt burnt at time t, and S as the surface area of the grain at this instant, it is possible to relate z to f or S to z. It is British practice to use (z, f) relation, while US ballisticians employ (S, z) relation. Either relation is known as the *form function.* See Ref for various equations employed
Ref: SACMS, InternalBallistics (1951), 40–52

FORMIC ACID AND DERIVATIVES

Formic or *Methanoic Acid* (Hydrogen Car-

boxylic Acid), H.COOH; mw 46.03; sp gr 1.2201 at 20/4°, colorless liq with pungent odor; fr p 8.6°, bp 100.8°, RI 1.3719 at 20°, fl p 156°F; it is dangerously caustic; sol in w, alc, eth & glycerol. Can be prepd by treating Na formate with sulfuric acid and distilling of the formic acid in vacuo; another method is acid hydrolysis of methyl formate. It is formed as by-product in the manuf of PE from formaldehyde and acetaldehyde in presence of slaked lime (Refs 2 & 3)

Used for prepn of formates, org esters, oxalic acid, allyl alc, fumigants, insecticides, refrigerants, lacquers, electroplating solns, and cellulose formate; also in silvering glass and as a rubber coagulant (Ref 5)

Refs: 1) Beil **2**, 8, (7) & [1] 2) Naoúm, NG(1928), 248 3) Davis(1943), 278 4) Kirk & Othmer **6**(1951), pp 875–83(43 refs) and 2nd Edn **10**(1966), pp 99–103 5) CondChemDict (1961), 511-R and **1971** Edn, 400-L

Azidoformic Acid Ethylester (Azidoameisensäure-äthylester, Triazoameisensäure-äthylester, Carbazidsäure-äthylester or Stickstoffkohlensäure-äthylester in Ger), $N_3COOCH_2CH_3$; mw 114.09, N 36.88%; liq, bp 114–15°, 34°(20.5 torr), sp gr 1.1082 at 20°, n_D 1.4162 at 25°. Prepd from N-aminourethane and nitric acid (Ref 2), or by reacting NaN_3 with chloroformic acid ethylester (Ref 3)

Refs: 1) Beil **3**, 129 & [101] 2) Hantzsch, Ber **36**, 2057(1903) 3) A. Bertho, JPrCh [2] **116**, 114(1927)

Azidoformic Acid Methylester (Azidoameisensäure-methylester, Triazoameisensäure-methylester, Carbazidsäure-methylester or Stickstoffkohlensäure-methylester in Ger), $N_3.CO.O.CH_3$; mw 101.06, N 41.58%, OB –55.4%; colorless liq, bp 102–3°; exploded occasionally when distilled under atm pressure. May be prepd by warming for 1 hr ethereal soln of Am azide with methyl ester of chloroformic acid, $Cl.CO.O.CH_3$.

Refs: 1) Beil **3**, 129 & [101] 2) T. Curtius et al, JPrCh [2] **52**, 480(1895) 3) A. Bertho, JPrCh [2] **116**, 112(1927)

Azidoformic Acid Dimethylamide, $N_3CON(CH_3)_2$; mw 114.11, N 49.10%; liq bp 59°(15 torr), expl when heated over a flame. Prepd by boiling NaN_3 with chloroformic acid dimethylamide in acet (Ref 2)

Refs: 1) Beil **4**, [575] 2) R. Stollé, JPrCh [2] **117**, 201(1927)

Ethyl Formate. See in this Vol, p E294-R

Glyceryl Formate. See in this Vol

Glyceryl Formate Dinitrate. See in this Vol

Glycol Formate. See in this Vol

Methyl Formate, $H.COO.CH_3$; mw 60.05; colorless, flammable liq with agreeable odor; sp gr 0.974 at 20/4°, fr p –99.8°, bp 31.8°, fl p –32°(–25.6°F), RI 1.3431 at 20°; sol in w, alc & eth. Can be prepd by heating methanol with Na formate and HCl, with subsequent distn. Used in org synthesis, cellulose acetate solvent, fumigant and for prepn of military poison gases

Refs: 1) Beil **2**, 18, (16), [25] & {28} 2) B.S. Lacy et al, JACS **52**, 928(1930) 3) CondChemDict (1961), 740-L; (1971), 574-L

Sodium Formate, H.COO.Na; mw 68.01; wh, sl hygroscopic powd; sp gr 1.919, mp 245° (dec); sol in w; sl sol in alc; insol in eth. Can be prepd by heating under pressure of NaOH with CO; purified by crystallization. Used as reducing agent, mordant and manuf of formic acid, methyl formate and oxalic acid

US Military requirements and tests for Sodium Formate for use as a reagent are in Specification MIL-S-11168(May 1951)

Refs: 1) Gmelin-Kraut Syst Number 21(1928), 806 2) Sax (1968), 1105-R 3) CondChemDict (1961), 1042-L; (1971), 801-L

Zinc Formate, $(HCOO)_2Zn.2H_2O$; mw 191.45; wh crysts, sp gr 2.158 at 21°; mp decomp at 175°; sol in w; insol in alc or eth. Can be prepd by the action of formic acid on Zn

hydroxide. Used as catalyst in manuf of methanol; also as waterproofing agent and wood preservative
Refs: 1) Gmelin-Kraut, not found 2) Sax (1968), 1245-R 3) CondChemDict (1961), 1245-R; (1971), 950-R

Formin or **Glyceryl Formiate.** See Formylglycerol in this Vol

Formit (Formite). One of the Ersatzsprengstoffe (Substitute Explosives) developed during WWII by an explosive group under the direction of Dr Hans Walter. It was prepd by heating a mixt of 30% commercial formaldehyde with Amm Nitrate (in the ratio 6 moles HCHO to 6 moles NH_4NO_3) under reflux for about 1 hour, followed by vacuum distillation to remove the water and unreacted formaldehyde. The residue was a faintly yellow composition which consisted of MAN-Salz 25 to 30, TRI-Salz 1 to 3 and AN 67 to 74%. Its calorific value was 900kcal/kg and volume of gases produced on explosion 1050 l/kg (calculated at 0° and 760mm Hg). When about 15% of RDX or PETN was incorporated, the velocity of detonation was increased appreciably and the brisance was increased to that of TNT, while the volume of gases evolved on explosion was higher than for TNT. This explosive could be cast-loaded (setting point about 90°) in projectiles, but unfortunately it exuded at 60–70°. It was fairly stable to heat provided no iron impurities were present
Note: *Man-Salz* is Ger name for Methylamine Nitrate, while *Tri-Salz* is for Trimethylammonium Nitrate
Refs: 1) H. Walter et al, "German Development in High Explosives", PB Rept **78271** (1947), p 4 2) A. LeRoux, MP **34**, 132 (1952) 3) Fedoroff et al, PATR **2510** (1958), p Ger 52

Formoguanamine or **2,4-Diimino-tetrahydro-1,3,5-triazine,** [HN=CNHC(=NH)NHCH=N], isomeric with **2,4-Diamino-1,3,5-triazine,** [H_2NC=NC(NH_2)=NCH=N], mw 111.10, N 63.38%; ndls (from w), mp 329° (dec); sl sol in boiling w; dif sol in alc; sol in dil HCl or warm dil sulfuric acid. Prepd by heating guanidine formate (Ref 2) or by treating biguanide hydrochloride with alcoholic KOH and chlf (Ref 3). Forms a mononitrate salt (Ref 2)
Refs: 1) Beil **26**, 225 & (65) 2) M. Nencki, Ber **7**, 1584 (1874) 3) E. Bamberger & W. Dieckmann, Ber **25**, 535 (1892)

Formula 226. See under Aluminum Containing Explosives, Vol **1**, p A146-L

Formulation of an Explosive. In order to compose an expl mixture suitable for certain kinds of work (mining, quarrying, ditching, stumping, etc), it is first necessary to know the props of the ingredients which one desires to use and the props required from such mixtures. For instance, in composing an expl suitable for coal mining it is necessary to determine what gases will be produced on expln and to calculate the total heat of combustion. This is done by adding the heats of combustion of ingredients and dividing by the average specific heat of the gases produced. This is called formulation. The formulation of expls is a highly developed art which has resulted in the development of blasting expls which are quite safe to manuf, transport and use, and which develop their strength in the particular way best adapted to the needs of the special use for which they are formulated

Formyl Explosive Compounds

N-(γ-Azidoformylbutyryl)glycine Azide (formerly called in Ger "Azidoglutarsäure-glycinazid"), $N_3CO(CH_2)_3CONHCH_2CON_3$; mw 239.21, N 40.99%, thick oil which expl when touched with a flame. Prepd by treatment of the corresponding dihydrazide with nitrous acid (Ref 2)

Refs: 1) Beil **4**, [791] 2) T. Curtius et al, JPrCh [2] **105**, 324(1922)

o-(Azidoformyl)phenylisocyanate (2-Isocyanato-benzoyl azide), mw 188.15, N 29.78%; ndls (from benz), mp 60° (dec) on careful heating, expl violently on rapid heating or on impact. May be prepd by heating phthalic acid diazide in benz (Ref 2)
Refs: 1) Beil **14**, [225] 2) H. Lindemann & W. Schultheis, Ann **464**, 250(1928)

N-(β-Azidoformylpropionyl)glycine Azide (formerly called in Ger "Azidosuccinylglycinazid"), $N_3CO(CH_2)_2CONHCH_2CON_3$, leaflets, mw 225.17, N 43.54%; expl when touched with a flame. Prepd by treating the corresponding dihydrazide with nitrous acid (Ref 2)
Refs: 1) Beil **4**, [791] 2) T. Curtius et al, JPrCh [2] **105**, 305(1922)

Formylhydroperoxide or **Performic Acid** (formerly Perameisensäure in Ger), HCO.O.OH; mw 62.03, OB 0.0%. Prepd as a 90% soln by distilling a mixture of formic acid, 100% hydrogen peroxide and sulfuric acid (Ref 2). The soln is more volatile than formic acid, is miscible with water, alc, eth and readily sol in benz or chlf. Metals and metal oxides may cause it to dec explosively. The soln is not stable even at 0° and can explode when shocked or heated
Refs: 1) Beil **2**, (19), [34] 2) J.D'Ans & A. Kneip, Ber **48**, 1137(1915) 3) Sax (1968), 1002-L 4) CondChemDict (1971), 669-R

1-Formyl-2-picrylhydrazine, $HCONHNH.C_6H_2(NO_2)_3$; mw 271.15, N 25.83%, OB −56.1%; mp 188.5° (dec). Prepd by the reaction of Picric Acid with "isodiazomethane", $(CH{=}N{-}NH)_x$, in eth, or by boiling picrylhydrazine with formic acid (Ref 2)

Refs: 1) Beil, not found 2) E. Müller & W. Kreutzmann, Ann **512**, 264–275(1934) & CA **29**, 769[5](1935)

Formylglycerol (or Formin) and Derivatives *Formylglycerol, Formin, Glycerol Monoformin* or *Glyceryl Formate,* $CH_2(OCHO).CH(OH).CH_2(OH)$, mw 120.10; liq, bp 154–57° at 10mm, d 1.3052 at 0°; n_D 1.4614 at 12.5° (Ref 1). Can be prepd by treating anhydrous oxalic acid with glycerol accdg to the equation:

$$\begin{array}{l} CH_2OH \\ | \\ CHOH \\ | \\ CH_2OH \end{array} + \begin{array}{l} COOH \\ | \\ COOH \end{array} = \begin{array}{l} CH_2OCHO \\ | \\ CHOH \\ | \\ CH_2OH \end{array} + H_2O + CO_2$$

Served for prepn of expl dinitrate
Refs: 1) Beil **2**, 24, (19), [33] & {48} 2) R. Delaby & P. Dubois, CR **187**, 768, 950(1928) 3) Naoúm, NG(1928), 197

Formylglycerol Dinitrate, Dinitroformin or **Glyceryl Formate Dinitrate,** $CH_2(OCHO).CH(ONO_2).CH_2(ONO_2)$; mw 210.10, N 13.33%, OB to CO_2 −22.2%; lt yel oily subst. Can be prepd by the action of nitric acid on the product of the reaction of glycerol and oxalic acid, which is glyceryl monoformin, $CH_2(OCHO).CHOH.CH_2OH$. It is an expl compd, more brisant and powerful than Dinitroacetin, described in Vol **1** of Encycl, p A35-L, under ACETINS

Formylglycerol Dinitrate was proposed by Dr Vezio Vender and used in Italy as an ingredient of low-freezing Dynamites (freezing below −20°). It is, however, more expensive than other antifreeze compds and has no advantage over them. Its mixt with NG(30:70) has 90% of the power of NG

Dinitroform decomps accdg to the equation: $CH_2(OCHO).CH(ONO_2).CH_2(ONO_2) = 2CO_2 + 2CO + 2H_2O + H_2 + N_2$, with evoln of 1009cal/g at C_v and H_2O liq. Its heat of expln is 68% that of NG
Refs: 1) Beil **2**, 24 2) Naoúm, NG(1928), 197 & 374 3) Davis(1943), 222

Formwar. A polyvinylformal insulating agent manufd by Bond Master Rubber Adhesive Co, Irvington, NJ, used in conjunction with bonding agent "Araldit" (epoxy resin of Ciba-Geigy Corp, Ardsley, NY) in some US Military detonators, such as T44
Refs: 1) P.B. Tweed, Ordn **44**, 654(1960) 2) P.B. Tweed, formerly of PicArsn; private communication (1960)

Forster (Von) patented at the end of the 19th century a smokeless proplnt prepd by treating with 2:1 ether-alcohol a mixture of 80% Guncotton with 20% Collodion Cotton. The resulting partially gelatinized product was rolled into sheets of 0.1mm thickness and cut into grains 2x2mm. As the surface of grains was rough, they could be dried quicker than grains with smooth surface obtd with completely gelatinized proplnts
Ref: Daniel, Dict (1902), 304 & 793

Fortex. Two expls patented in France in 1910 were:
Fortex No 1: NH_4NO_3 70 & Tetryl 30 and *Fortex Antigrisouteux:* NH_4NO_3 78.5 & Tetryl 21.5%. Calculation showed that OB to CO_2 of the first expl was practically zero, while for the second there was an excess of oxygen. They were not authorized by the Commission des Substances Explosives (French Explosives Commission)(Ref 1).
A new Fortex is one of the few commercial expls based on Tetryl: Tetryl 12, Amm Nitrate 35, K nitrate 33 & Amm chloride 20 (Ref 3)
Refs: 1) H. Dautriche, MP **17**, 177–79 (1914) & CA **8**, 1208(1914) 2) Colver (1918), 373 3) Thorpe **4**, 487(1940) 4) Clift & Fedoroff **2** (1943), p F3

Fortification. A structure or earthworks, usually heavily armed, constructed as a defense. Also – a fortified place or position
Ref: Glossary of Ordn (1959), 122-L

Fortification or Revivification of Acids. In the manuf of expls, such as NC, NG, TNT, PETN etc by nitration, the main reaction results in the consumption of a certain quantity of nitric acid and the formation of an equivalent quantity of water. The resulting spent acid is usually too weak for nitrations, but in some cases it may be fortified (in works' parlance, "butted-up") by the addition of concd nitric acid (60–99%) and oleum or by a suitable strength mixed acid

Sometimes, for example, in the nitration of toluene to Mononitrotoluene, the spent acid contains so much water and so little nitric, that it is impossible to fortify economically with nitric acid and oleum. In this case the acid (commonly called "waste acid") has to be distilled in order to recover the nitric acid, and the remaining sulfuric acid is concd to about 95% strength
Examples of Calculation. Assuming that a spent acid ("di-spent") left after the dinitration of toluene with a composition which is as follows: total H_2SO_4 74.6, total HNO_3 12.6 (actual HNO_3 plus oxides of N calcd as HNO_3), H_2O 14.7, and that it is desired to fortify it to obtain a mixture with the composition: total H_2SO_4 66.3, total HNO_3 21.4 and H_2O 7.8%. The calculation would proceed as follows: to the waste acid add a volume of a mixture containing $(Qx'-x)$%, each of H_2SO_4, HNO_3, and H_2O, wherein x' is the desired %, x is the % in the waste acid and Q is the ratio of the new total volume to the volume of waste acid

The above described method of calcn was used during WWII at the Keystone Ordnance Works, Meadville, Pa

Explanation of terms total H_2SO_4, total HNO_3, actual HNO_3, etc is given in Vol 1 of Encycl, under "Acidity in Acids", pp A88-R to A90-R

Fortis. Accdg to Daniel (Ref 1, p 304) it was a powder which is also known as *Polynitrocellulose, Poudre d'Heusschen, Glyceronitre* or *Benzoglyceronitre.* The

Fortis No 1, patented in England in 1884, had the following compn: K nitrate 57.00 to 93.19, sulfur 8.00 to 12.00, tanbark 15.00 to 25.00, lampblack 2.00 to 3.00, Fe sulfate 1.35 to 27.80 & glycerol 0.95 to 1.84%. The *Fortis 2* contd besides the above, naphthalene and Na nitrate. It was claimed that when these mixtures were initiated, some NG was produced right in the boreholes

The following *Fortis* was patented in France in 1891 (Ref 1, p 305): K or Na nitrate 65, sulfur 13, carbon 12 & DNNaphthalene 10%. The same expl is listed by Gody (Ref 2), but %age for DNN is given as 12%, which is evidently wrong because the total makes 102% instead of 100%. This *Fortis* was manufd by the SA des Poudreries Belges at Hérenthals and was admitted to England

Refs: 1) Daniel (1902), 304–05 2) Gody (1907), 173

Fortisine. A mixture of K nitrate 74.8, charcoal 14.4, sulfur 6.3, DNB 4.1 & moisture 0.4%

Refs: 1) Daniel (1902), 305 2) Fedoroff & Clift 4(1946), 36

Forty-Degree Test for Exudation (Sweating) of Explosives. This test is known in France as "Exsudation par étuvage". It is described in Vol 1 of Encycl, p XI

A similar British test, known as "Keeping Test" is described in Ref 1

The American *Forty-Degree Test* is one of the tests conducted at the Bureau of Mines, Pittsburgh, Pa for determining whether a Dynamite exudes or not. Although the official US Bur of Mines test is the *Centrifuge Test* (described in Vol 2 of Encycl, p C31-R), other tests may be used especially if there is no centrifuge available

Following is the procedure for Forty Degree Test:

1) With the aid of a thin aluminum wire, make several small perforations thru the wrapper on one end of the Dynamite cartridge

2) Place the cartridge upright on the perforated end in a small wire tripod located inside a tall glass beaker or a glass cylinder

3) Place the ensemble into a constant temp oven maintained at 40°

4) At the end of six days, remove the cartridge and examine to determine if any drops of NG or other liquid have formed at the perforated end

If drops are found, the expl is considered to contain too great a percentage of NG, or other liquid ingredients, for the amount of absorbents present and the composition should be changed accordingly

Refs: 1) Marshall 2, (1917), 421 2) C.E. Munroe & J.E. Tiffany, "Physical Testing of Explosives", Bur of Mines Bull 346, Washington, DC (1931), p 26

Forty-foot Drop Test. A complete description of the procedure is given as test No 103 of MIL-STD-331. See Vol 4, p D1093-R, Test f3

Forty Percent Dynamite. See Farmer's Dynamite

Forty-two Grain Powder. A sporting shotgun powder of such apparent density that 42 grains are required as a charge for a 12-bore cartridge, in order to occupy the same space as 82 grains of Black Powder

Schultze, Amberite, Ruby, Felixite, Primrose Smokeless, Coopal No 1, KS Powder etc belonged to this class of powder. Some of the 42-grain powders are (probably) still used occasionally, but the powders for which smaller charges are required (such as 33-grain powder) are preferred because they give lighter recoil and burn quicker. Reduction of charge is effected by using a NC of higher nitrogen content and reducing the proportion of other ingredients. These changes increase the rate of burning which might result in the development of dangerous pressures in the gun. In order to slow down these

powders, more complete gelatinization of the NC is required
Ref: Marshall **1**, (1917), 326

Fossano (Polvere). One of the older Italian Black Powders
Ref: Daniel (1902), 305

Fossano Powder. A progressive burning powder which was prepd as follows: Black Powder, as it comes from the mill("mill cake") was pressed to a density of 1.79 and then broken into 1/8" to 1/4" grains. These grains were mixed with fine Black Powder and the resulting mixture pressed into grains 2½" x 2½" x 1¾" with an average density of 1.76. This powder may be considered as a development of "Doremus Cartridge", described in Vol **5** of Encycl, p D1535-L
Ref: Van Gelder & Schlattek (1927), 26

Fossil Flour. Same as kieselguhr

Fougas or **Fugas** (Rus). A kind of landmine prepd by burying several feet underground a large charge of explosive (such as BkPdr), placing on top of it heavy stones or metallic objects, and covering the ensemble with earth. The chge of expl was detonated by fuse or by electric cap when a vehicle or body of troops approached or went over the fougas

Fougas can also be placed in tunnels dug underground towards enemy's fortifications and then exploded under them. This is done in order to make an opening (breach) in the wall (called in Russ "bresh") or to destroy the fortification gate
Ref: F. Pavlenkoff, "Entsiklopedicheskii Slovar" (Encyclopedic Dictionary), St Petersbourg, Russia (1913), p 2770 (Reproduced by Bookstore N.N. Mart'ianov, 243 W56 St, New York 10019

Fougasse (Fr). A kind of land mine prepd by digging underground an inclined hole in the form of truncated pyramid and burying in it an hermetic box contg explosive. This was covered with a template, heavy stones, and earth. Detonation of expl chge was done either by fuse or by electric cap
Ref: Daniel, Dict (1902), 307–08

Fougasse, Incendiary Fuels for. Exptl work on fillings for fougasses led to the following compn: 60 gals gasoline, 55 gals rubber latex, 40 gals alc, 25 lbs stearic acid and 7 pts NaOH soln of sp gr 1.43
Ref: J InstPetroleum **34**, 90–108 (1948) & CA **42**, 3961 (1948)

Fougasse (US). Accdg to definition given in Glossary of Ordn (1959), p 122-L, it is a mine constructed so that upon expln of the charge, pieces of metal, rock, gasoline or other substances are blown in a predetermined direction

Fouling of Guns and Antifouling Agents.

The advent of smokeless proplnts required the coating of the lead bullet, normally with a Cu-Ni alloy. With repeated firings some of this alloy builds up inside the gun barrel, trapping acidic residues from the proplnt and primer. The result is severe corrosion of the barrel and interference with the ballistics of the bullets from increased friction. Preventive measures involved the incorporation of various metal scavengers into the propelnt as exemplified in Refs: 1) 1–5% Pb or Sn oxide or carbonate; 3 & 4) aromatic or aliphatic Sn compounds; 5 & 6) 0.25–2.0% Sb oxide or sulfide, Cd metal, oxide or carbonate, Co, MnO_2, ground glass or calcium silicide; 7) CaH_2. Barrel cleaning formulations based on an alkaline oxidizing agent were typified by Refs: 2) aqueous $(NH_4)_2SO_4$, $(NH_4)_2S_2O_8$, NaOH – dissolved just before use; and 8) aqueous NH_4OH, oleic acid, $K_2Cr_2O_7$ and H_2SO_4, in de-

creasing order

One of the devices used occasionally by the Russians for preventing excessive fouling of gun barrels was a lubricator, called *prossal'nik* ("lubricating gasket" in Engl and "Dichtungsdeckel" in Ger). Accdg to a brief description given in Ref 9, the device consisted of cardboard obturator, cardboard covers (between which was placed a thick ring consisting of a lubricating mixture of red lead, ceresin and vaselin) and a gasket. It was usually mounted on the boat tail (or base) of the projectile during loading. It was claimed that proplnt gases caused the "prossal'nik" to disintegrate and the lubricating material to be spattered inside the bore, thus depositing a protective insulating coating on the bore surface

It was claimed by the Russians that "prossal'nik" increased the life of the guns, but accdg to some American sources, the use of bore lubrication decreased the muzzle velocity

Accdg to J.E. Capell, formerly of Pic-Arsenal, some other (than US or Russia) countries tried friction lubricants, but they did not think that they were of great value (See also Decoppering Agents in Vol **3**, p D36-R)

Refs: 1) C. Henning, USP 1357865 (1920) & CA **15**, 315 (1921) 2) R. Laughlin, USP 1535124 (1925) & CA **19**, 1949 (1925) 3a) Imperial Chemical Ind Ltd, FrP 708855 (1931) & CA **26**, 1125 (1932) 3b) Ibid, FrP 730728 (1932) & CA **27**, 419 (1933) 3c) Ibid, BrP 373152 (1931) 4) R. Burns & O.W. Stickland, USP 1943421 (1934) & CA **28**, 2188 (1934) 5a) H.N. Marsh, USP 2131061 (1938) & CA **32**, 9504 (1938) 5b) Ibid, USP 2131352 (1938) & CA, Ibid 5c) Ibid, USP 2131353 (1938) & CA, Ibid 5d) Ibid, USP 2131354 (1938) & CA, Ibid 5e) Ibid, USP 2131383 (1938) & CA, Ibid 6) Hercules Powder Co, BrP 508914 (1939) & CA **34**, 3092 (1940) 7) V.N. Poddubny, TekhiVooruzhenie **1938**, 60–7 & ChemZentr **1938**, II, 1480 & CA **34**, 5037 (1940) 8) C. Hardy, USP 2301043 (1942) & CA **37**, 2185 (1943) 9) Fedoroff et al, PATR **2145** (1955), p Rus 19-L

Four Cartridge Test. German test designed to determine the ability of mining explosives to transmit detonation. It is called **Detonationsfähigkeit Probe** and was conducted as follows:

Four cartridges, 35mm in diam, were laid end to end on a bed of sand and then one side of the train was detonated by a No 3 Blasting Cap. It was required that all four cartridges be detonated completely

It is one of the "Detonation by Influence Tests", listed in Vol **1**, p X and in Vol **4**, pp D395-R to D402-L. The most important of these tests are "Gap Tests". They are briefly described in Vol **1**, pp XIV & XV and listed in Vol **4**, pp D303-R & D304-L. The LSGT (Large-Scale Gap Test), designed by Donna Price, is described in Vol **4**, pp D318-R & D319-L. The NOLR (Naval Ordnance Laboratory) Gap Test is on pp D321 & D322-L and its modification on p D327, Fig 8. The SSGT (Small Scale Gap Test) is briefly described on pp D318 and more fully by J.N. Ayres in NavWepsRept **7342** (1961) and by Donna Price in NOLTR (1966), which is abstracted on pp D343-R to D345-L with Fig 35 on p D344

Refs for German Test: 1) BIOS Final Rept **1266** (1942), p 2 2) Fedoroff et al, PATR **2510** (1958), p Ger 52-R

Fourcroy-Vaquelin Detonating Mixture, proposed in France about 1790, consisted of powdered potassium chlorate mixed with powdered sulfur. Calcium or sodium chlorate in lieu of K chlorate were also tried. All of these mixts were extremely dangerous to handle

Refs: 1) M. Specter, SS **26**, 372–73 (1931) 2) Ibid, SS **27**, 332 (1932)

Fournier Powder (Brit). Patented in 1870; was prepd by heating a mixture of 125 parts Ca carbonate and 65 ps of NaCl, all well covered with urine. After evaporating nearly to dryness, 35 ps of carbon was added and the mass well mixed

Refs: 1) Daniel (1902), 308 2) Fedoroff & Clift 4(1946), 36

Fowler Dynamite. One of the Ital "dinamiti a base attiva": NG 20.00, AN 56.25, carbon 5.00 & Ca sulfate 18.75%
Refs: 1) Daniel (1902), 305 2) Giua, Trattato VI(1)(1959), 340

Foxhole. Small pit used for cover of one or two soldiers. It is so constructed that an occupant can fire effectively from it
Ref: Glossary of Ordn(1959), 122-L

Foxhole Explosive Digger. The Harve Machine Co design consists of a finless rocket with a 1-3/8-inch motor containing 0.18 lbs of a double-base proplnt and a head contg 0.56 lbs of HBX. The rocket head is detonated by a vane type fuze, the vanes of which are turned by the earth as the rocket penetrates. Suspended from the front of the rocket is a small shaped chge that is fired by the fuze ogive upon the initial movement of the rocket

The whole assembly is suspended near its center of gravity from a simple lightweight tripod launcher. The assembly is folded and inserted into a water proof tubular fiberboard container and the legs of the launcher are used to hold the rocket and shaped chge securely in the packed condition. Also one leg carries a safety pin for the fuze while a second carries a leg extension

The rocket is fired by an M2 Fuse and about 16 inches of safety fuse

It should be noted that the Rocket-Shaped Charged-Launcher combination comes as a complete assembly and needs no field assembly or disassembly. This item is intended for one use only – a one shot cratering device. It must be emphasized that the Explosive Foxhole Digger, if properly used, can be a valuable tool, but if not properly used, can be a hazardous expl item. All possible safety has been designed into the item, but nothing can render it safe from intentional misuse
Ref: Staff, Harve Machine Co Inc, Torrance, California, "Characteristics and Operation of Harve Machine Co, Inc Design Explosive Foxhole Digger" (June 1957) (Contract DA-44-009-Eng-2906 for Corps of Engineers, Fort Belvoir, Va)

FP. Amer abbr for "Flashless Propellant"

Fp. Ger abbr for *Füllpulver* which is any high explosive used for filling shells, bombs, mines, torpedoes or grenades. It may be called *filler* or bursting charge

Fp 02 (Füllpulver 02). Explosive Pattern 1902 or TNT

Fp 88 (Füllpulver 88). Explosive Pattern 1888 or PA

Fp 60/40 (Füllpulver 60/40). Amatol contg TNT 60 & Amm Nitrate 40%
Ref: PATR **2510**(1958), p Ger 52-R

fps. Amer abbr for "feet per second"

Fractionation Columns (Towers or Tubes) are devices used in fractional distillation (See also under DISTILLATION in Vol **5**, p D1510-L)

The most commonly used types of columns are: plate columns, Vigreux columns, packed columns (packed with such materials as Raschig rings, Berl saddles, fibrous glass etc)
Refs: 1) C.S. Robinson & E.R. Gilliland, "The Elements of Fractional Distillation", McGraw-Hill, NY (1939) 2) A. Rose, IEC **33**, 594 (1941)(Batch fractionation) 3) D.F. Othmer, ChemMetEng **49**, 84(1942)(Distillation practices and methods of calculation) 4) G.W. Minard et al, TransAmInstChemEngrs **39**, 813

(1943)(Fibrous glass as a packing material for packed column distillation) 5) E. Riegel, "Chemical Machinery", Reinhold, NY (1944), pp 388–395 6) VanNostrand Chemist's Dictionary (1951), 303-R 7) Perry (1963), 13-2

Fractorites. European Dynamites, listed in Table F13 are taken from the book of Gody (Ref 1, pp 712, 713 & 715). The 1st and 3rd compns are also listed in Thorpe (Ref 2) and in Ref 4. The 3rd compn is also listed by Naoúm (Ref 3)

Table F13
Fractorites

	Designation			
Components	? Matagne	B Matagne	D Matagne	CA SAPRB
AN	90	75	75	Other ingredients were not given in Ref 6
Colophony	4	–	–	
Dextrine	4	–	–	
K dichromate	2	–	–	
Amm oxalate	–	2.2	7	
Amm chloride	–	20	–	
DNN(Dinitro-naphthaline	–	2.8	–	
Na nitrate	–	–	10	
NG	–	–	4	10
Wheat flour	–	–	4	

Refs: 1) Gody (1907), 703, 705, 712, 713 & 715 2) Thorpe **2**(1917), 416 3) Naoúm, NG (1928), 439 4) Clift & Fedoroff **2**(1943), p F3 5) Anon, Explosifs (Belg) No **4**, (1958) p 64 6) L. Deffet & F. Lebrun, Brussels; private communication (1960)

Fracturing by Relative Motion of Load in Detonation. See Vol **4**, p D544-R

Fracturing by Release of Load in Detonation. See Vol **4**, pp D543-R & D544-L

Fracturing by Shear in Detonation. See Shear Fracture in Vol **4**, p D544-R. The method has been used in tunnel driving under the name *Burn-Cut Round Method* (p D545-L)

Fracturing in Oil Shale by Explosives. BuMines developed methods for fragmenting oil shale formations with chemical explosives to prepare oil shale for in situ retorting and to evaluate the extent of fragmentation by various methods. A combination of three methods of explosive fracturing was used: (1) The displacement and detonation of a liquid chemical explosive in a natural fracture system; (2) the use of 60-pct dynamite to relieve stress conditions of the rock around the wellbore; and (3) the use of pelletized TNT in a series of wellbore shots to fragment the oil shale. The data from the seismic measurements indicated the extensively fragmented zone was approx 95 ft in diameter and 70 ft thick
Refs: 1) J.S. Miller & H.R. Nicholls, "Methods and Evaluation of Explosive Fracturing in Oil Shale", USBurMines Report of Investigation **RI 7729**(1973), Pittsburgh, Pa, 19213 2) G. Cohn, Edit, Expls&Pyrots **7**(2), 1974

Fracturing of Corner in Detonation. See Vol **4**, p D543-R

Fracturing of Metal in Detonation or Fracturing of Metal Under Impact Loading by High Explosives. See Vol **4**, p D542-R

Fracturing of Rock by Shock-Wave in Detonation. See Vol **4**, p D545-R and Table 2 on p D546

Frag. Abbr for Fragment or Fragmentation

Fragmacord Mine. A new concept of linear A/P (antipersonnel) mine which combines heavy detonating cord with a fragmenting

system, consists of a 25-foot length of flexible expl cord 1/2 inch in diam, tightly bound at 1/4 inch intervals with metal rings notched to break into at least 12 fragments each when the cord is detonated. Thus, from a 25-foot length, at least 3000 fragments are produced. The new mine is versatile, being easily portable and quickly emplaced, and hence was well suited for such unconventional combat situations as were commonplace in Vietnam
Ref: R.T. Schimmel & S.J. Lowell, PATR **3375**(1966)

Fragment. A piece of an exploding or exploded bomb, projectile or the like
Ref: Glossary of Ordn (1959), 122-L

Fragmentation. Term applied to ammunition, indicates that the item is primarily intended to produce a fragmentation effect
Ref: Glossary of Ordn (1959), 122-L

Fragmentation Bombs are described in Vol **4**, pp D933 to D935. One of such bombs, *90-lb Frag Bomb*, M82 is shown in Fig 2-2 on p D935

Fragmentation Characteristics. As a manifestation of expl output fragmentation is characterized by velocity and size distribution of fragments. For some purposes, the size and shape of fragments are predetermined either by preforming or by modifications of the case or chge design which predisposes it to break as desired

The initial velocity of fragments is quite accurately predicted by the Gurney formulas described by us under "Gurney Constant and Gurney Formulas" in Vol **6** of Encycl

More detailed discussion on "Fragmentation Characteristics" is given in:
Ref: Anon, "Engineering Design Handbook, Explosive Trains", US Army Materiel Command Pamphlet **AMCP 706-179**(1974), pp 3-14, 3-15 & 3-16

Fragment Concentration Test. See Density of Fragments (Splinters) Test in Vol **3**, p D84-L and in PATR **2510**, p Ger 52-R

Fragmentation, Controlled. Since the breakup of chge cases under expl attack is mainly two dimensional, the average size of fragments may be reduced and their number increased by the use of multiple walled cases. Many other methods were developed to produce fragments that are almost all of the optimum size and shape. These methods include:

a) Preformed fragments with or w/o matrix
b) Notched or grooved rings
c) Notched or grooved wire
d) Notched or grooved casings
e) Fluted liners
f) Preformed rod fragments. They are more effective against aircraft than the same weight of metal broken into smaller pieces. There are two systems:

Discrete rod warhead consists of a number of rods (usually of steel) arranged like the staves of a barrel to form a cylindrical container which is provided with covers. The rods are joined together to provide a needed structural strength for handling, launching and flight but not to prevent their separation under the action of the expl, which is placed inside the container

Continuous rod warhead consists of rods that are strongly joined to one another at alternate ends in a manner similar to that of a folded carpenters' rule. The hoop breaks when its circumference equals the sum of the rod length, if excess energy is imparted by expl
Ref: AMCP 706-179(1974), p 3-16

Fragment Density Test. Same as Fragment Concentration Test

Fragmentation Grenades, such as: *Fragmentation Hand Grenades.* See Vol **4**, pp D830 & D831 and Figs 1-20a, 1-20b & 1-20c

When grenades are fired from rifles, they are called *Rifle Grenades.* They can be either especially designed or adapted to be fired or launched from the muzzle of a rifle or carbine
Ref: Glossary of Ordn (1959), 138-39

Fragment Gun. See Vol 1, p XII

Fragment Gun Test (FGT). See under Fragment Gun. The FGT values for several expls are given under BRISANCE in Table 1 of Vol 2, pp B266 to B295

Fragmentation Tests in Detonation and Explosion. These tests were designed to determine the actual performance (efficiency) of an expl projectile or any of its parts. They permit the approx estimation of the brisance of the expl used in the projectile. Refs for these tests are given in Vol 1, p XII and a list of various fragmentation tests is given in Vol 4, p 303-L, under Experimental Procedures. The so-called Closed Pit and Open Pit Tests are described in Vol 3, pp C345-R to C350-L. For other information, see individual compns; eg Comp B Table, Vol 3, p C479, etc
Addnl Refs: 1) Stettbacher (1933), 50–51, 218–19 2) L.V. Clark, IEC **25**, 389–90 (1933) 3) A. Majrich & F. Sorm, SS **30**, 298–99 (1935) 4) A. Stettbacher, Kriegssprengstoffe, Protar **8**, 90 (1942) 5) D.R. Cameron, PATR **1400** (1944) 6) W.R. Tomlinson Jr, PATR **1404** (1944) 7) W.R. Tomlinson Jr, PATR **1590** (1945) 8) S. Fleischnick, PATR **1595** (1946) 9) N.R. Mott, "Fragmentation of Shell Cases", PrRoySoc **A189**, 300–08 (1947) 10) Globe American Corp, Kokomo, Ind, "T28ET Fragmentation Bomb Clusters", ProgrRept **20** (Nov 1954) (Contract No DA-33-008-ORD-618) 11) Anon, "Military Explosives", **TM9-1910** (1955), 63–5 12) M.M. Jones & O.E. Sheffield, "The Sand Bomb Test Considered as a Fragmentation Problem", PATR **2424** (1957) 13) L.D. Heppner, "A Fragmentation Test to Improve Fragment Velocity Data", ABG Misc Rept 299 (1959) 14) Anon, "Military Explosives", **TM9-1300-214** (1967), 5–18

Fragment Velocity Measurements. In addn to a brief description given in Vol 1 of Encycl, p XIII, the following may be included:

The faster the fragments of a bomb or a shell fly, the more damage they might cause to a target. It is important for this reason to be able to determine their velocities and this info will be useful in detg the relative brisance of the expl with which the projectile is filled

The US Ordnance method employed during and after WWII for detg fragment velocity utilized high-speed motion cameras, which could be operated at speeds as high as 14000 frames per second. The test has been conducted (behind a barricade) by suspending the round of ammunition at a certain measured distance from metal (such as "dural") targets to be photographed, located near the fragment recovery boxes. A mirror was placed to reflect an image of the round to the camera

When prepns were completed, the cameras were started and the round fired. If the velocities of fragments were higher than 1700–1800fps, light flashed results from fragments impacting upon the metal target and these flashes were recorded by camera. By knowing the distance from round to target and time elapsed, it was possible to determine the velocity. After the firing was over, the testing personnel returned to the scene in order to collect the fragments, record the location of each fragment on the target and to weigh each fragment

For more info see Refs of which Refs 1 & 6 describe methods for detg fragment velocities using argon light chronograph (See also "Guerney Constant" and "Guemey Formulas" in this Vol and in Refs
Refs: 1) ARD (Armament Research Dept) Explosives Report **31/43**–AC 3430–SD/FP74

(1943) 2) R.W. Gurney, "The Initial Velocities of Fragments from Bombs, Shells and Grenades", RRL **405**(14 Sept 1943) 3) OSRD Rept **5746**(1945) 4) OSRD Rept **5622**(1946) 5) Armament Design Establishment (ADE) Report **9/51**(1951) 6) ARD Rept **19/51** (1951) 7) Ordn Proof Manual No **40-17-2** (1952) 7a) Baum, Stanyukovich & Shekhter (1959), 458-64 8) Ordn Proof Manual OPM **80-16**(1963) 9) AMCP **706-177**(1967), p 7-8 10) Materiel Test Procedure, MTP **4-2-814**(1967), superseding Ref 8 11) AMCP **706-179**(1974), p 3-15 (Guemey Constant and Guemey Formulas)

Framing Camera. See Vol **2**, p C14-R under CAMERAS

Franchimont, A.P.N. (1874-1914). Fr scientist who for 40 yrs was professor of org chem at Leiden University, Holland. The great numbers of students at his jubilee are witness that not only have many chemistry students been encouraged to further study by his teachings, but he left his impressions on many medical & pharmaceutical students. He built his contributions to chem on the work of his teacher, F.A. Kekulé, German scientist, at the University of Bonn. Professor Franchimont instituted lab work & teaching methods for beginning students in his textbook, "Introduction to Practical Exercises in Organic Chemistry for Beginning Students", Leiden (1897)

He is known not only for his teaching but also for his research, some of which had practical technical importance, such as his work on cellulose acetate & nitric acid. Professor Franchimont held honorary membership in the Chemical Society of London (1898), the Royal Institution of Great Britain & Ireland (1899), the Order of the Dutch Lions, and the Legion of Honor (Refs 1 & 2)

Refs: 1) H.J. Backer, ChemWeekblad **11**, 382-91 (1914) & CA **9**, 2871 (1915) 2) W. Adriani, ChemWeekblad **16**, 980-83 (1919) & CA **13**, 2157 (1919)

Franchimont Reaction. By the action of KCN on α-bromo acids or esters, α-cyano-α,β-dicarboxylic acid derivatives are formed which on hydrolysis and decarboxylation yield α,β-dicarboxylic acids:

$2C_6H_5CHBr.COOR + KCN \rightarrow$

$C_6H_5.C(CN)-COOR$ $\xrightarrow{H_2O}$ $C_6H_5.CH.COOH$
$C_6H_5.CH-COOR$ $\rightarrow$ $C_6H_5.CH.COOH$

Ref: The Van Nostrand Chem Dict (1951), 303-R & 304

François Blondel (1617-1686). A Fr ballistician who published in 1685 a treatise entitled "L'Art de Jetter les Bombes"

Ref: A. Basset, MAF **10**, 612-21 (1931)

Frangible Ammunition (Frangible Bullets and Frangible Projectiles). Frangible bullets are described under BULLETS in Vol **2** of Encycl, pp B331-R & B332-L and in Refs 1 & 3

Under the title *Frangible Projectiles*, Welden patented (Ref 2) projectiles contg 75-90% finely divided metal filings and 25-10% thermoplastic resinous binder. Such projectiles (bullets) were suitable for use in .22 cal cartridges generally used in shooting galleries and for other types of gunnery training. They eliminated many of the hazards accompanying the use of the lead projectiles which are commonly employed

Refs: 1) Staff, ArmyOrdn **29**, p 54 (July-Aug 1945) 2) L.C. Welden, USP 2593637 (1952) & CA **46**, 6838 (1952) 3) Anon, "Small Arms Ammunition", TM**9-1305-200**(1961), 46-7

Frangible Hand Grenade. Improvised incendiary hand grenade consisting of a glass container filled with a flammable liquid and provided with an igniter. It breaks and ignites upon striking a resistant target, such as a tank. The so-called "Molotov Cocktail" consisted of a glass bottle filled with a flammable liquid such as gasoline or benzene. After closing the bottle, a piece of fabric was wrapped around its neck. Before throwing the grenade, the fabric was soaked with a concd soln of white phosphorus in carbon disulfide.

As soon as part of carbon disulfide evaporated, P on coming in contact with air ignited and its flame ignited the flammable liquid of grenade

Another type of frangible grenade was filled with a mixture of K chlorate 80 & Nitrobenzene 20%, called "Russische Coctail" (Russian Cocktail) by Stettbacher. This mixt ignited on being touched with a drop of concd sulfuric acid. The reaction proceeded as follows:

$25KClO_3 + 6C_6H_5NO_2 =$
$36CO_2 + 15H_2O + 25KCl + 6N_2$
generating heat equal to 1182kcal/kg

Refs: 1) A. Stettbacher, Protar(Swiss) **10**, 160(1944) 2) Fedoroff et al, PATR **2145** (1955), p Rus 20-R 3) Glossary of Ordn (1959), 138-R(Grenade, Frangible)

Frank, Adolph (1834–1916), German industrial chemist who developed the potash industry
Ref: Hackh's (1944), 355-L

Frangible Projectiles. See under Frangible Ammunition

Franke patented in France in 1886, mixtures of nitrated benzene, phenol, naphthalene or naphthol with solid oxidizers, using as binders non-explosive substances, such as drying oils, soln of collodion or of gum, etc
Ref: Daniel, Dict (1902), 309

Frankel patented in France in 1888 & in 1899 powders consisting of K(Na, NH_4 or Pb) nitrate impregnated with a molten hydrocarbon (such as a mixt of naphthalene with paraffin). Such mixtures were activated by addn of powdered K chlorate just before use
Refs: 1) Cundill(1889) in MP **5**, 338(1892) 2) Daniel(1902), 308

Frank Explosives. Ammonium Nitrate explosives, patented in 1895 in Germany, contd as an additive K permanganate and/or chromate or bichromate. This was done in order to increase the power and sensitivity to initiation and to decrease the hygroscopicity

For example, if to an expl with low temp of expln and low sensitivity to initiation, such as the one contg 96% AN and 4% MN-Naphthalene, was added 4% K permanganate, the power, as detd by Trauzl Test, was nearly doubled and its sensitivity was increased to the point that it was possible to use for initiation No 3 cap (0.53g MF) instead of No 6 cap (1.0g MF)

Refs: 1) Daniel(1902), 308–09

Frankford Arsenal. One of the US Arsenals listed in Vol 1, p A490-L. It is a field installation of the Army Materiel Cmd located in Philadelphia, Pennsylvania charged with the responsibility for development, procurement and associated activities for off-carriage fire control systems, and on-carriage fire control components; also has responsibilities with respect to small arms ammunition, artillery cartridge cases, projectiles & mechanical time fuzes, standard inspection gages & measuring equipment, and metallurgical research & engineering. Also responsible for development of recoilless rifles & ammunition

Refs: 1) Glossary of Ordn(1959), 122-R 2) Anon, AMCP **70–1**(Feb 1968), "AMC RDTE Facilities and Capabilities", 497–550

Frank-Kamenetskii Thermal Explosion Theory. A thermal explosion theory as developed by D.A. Frank-Kamenetskii (Ref 5) led to the equation $\Delta T = -(Q/\lambda) z e^{-E/RT_o} e^{(E/RT_o^2)(T-T_o)}$. The ignition temp expln limits so calcd agree with those experimentally found for azomethane by Rice (Ref 1), $MeNO_2$ by Appin, N_2O by Zel'dovich and Yakovlev (Ref 4); and for H_2S by Yakovlev and Shantarovich (Ref 3). The expl limit calcd for N_2O was subsequently found experimentally. With Et azide, the calcd

values disagree with the exptl values of Rice and Campbell (Ref 2). Also See Vol **4**, pp D408-R & D409-L

Refs: 1) O.K. Rice, ProcNatAcadSci **14**, 113–18 (1928) & CA **22**, 1889 (1928) 2) H.C. Campbell & O.K. Rice, JACS **57**, 1044–50 (1935) & CA **29**, 5273 (1935) 3) B. Yakovlev & P. Shantarovich, ZhFizKhim(Rus) **9**, 112–31 (1937) & CA **31**, 4191 (1937) 4) Ya.B. Zel'dovich & B.I. Takovlev, DoklAkadNauk-(Rus) **19**, 699 (1938) & CA **32**, 9502 (1938 5) D.A. Frank-Kamenetskii, ActaPhysicoChim-(Rus) **10**, 365–70 (1939) & CA **33**, 6049–50 (1939)

Frankland, Sir Edward (1825–1899). A British chemist noted for research on organic-metallic compds, valency, water supply and the theory of flames. Discovered, in collaboration with Brit astronomer Sir Joseph Lockyear (1836–1920), helium in the sun's chromosphere

Ref: Hackh's Dict (1944), 355-L & 498-L

Frankland Reaction is the synthesis of hydrocarbons by zinc-alkyl condensation:

$$Zn(CH_3)_2 + 2R.Br = 2R.CH_3 + ZnBr_2$$

Ref: Hackh's Dict (1944), 355-R

Franklin, Benjamin (1706–1790). An Amer statesman, printer, scientist and writer, born in Boston; went to Philadelphia in 1723. Architect of the Declaration of Independence and Constitution. Minister to France. First postmaster. Known internationally for his common-sense philosophy. For details, see his writings or any Encycl. Inventor of elect initiation (Ref 1)

He founded the American Philosophical Society and developed some fertilizers (Ref 2)

Refs: 1) C.E. Munroe, "Benjamin Franklin's Unheralded Achievement", ExplosivesEngineer **3**, 115–18 (1925) 2) Hackh's Dict (1944), 355-R

Franklin, Edward C. (1862–1937). An Amer chemist noted for his theory of the *ammonia system compounds* (Ref, p 355-R)

It is a system of acids, bases and salts with liquid ammonia as solvent instead of water. Thus the *water system:*

$$\underset{\text{aquo -base}}{MOH} + \underset{\text{aquo -acid}}{HX} \underset{\text{hydrolysis}}{\overset{\text{neutralization}}{\rightleftharpoons}} \underset{\text{aquo -salt}}{MX} + HOH$$

and in the *ammonia system:*

$$\underset{\text{ammonio -base}}{M.NH_2} + \underset{\text{ammono -acid}}{HX} \underset{\text{ammonolysis}}{\overset{\text{neutralization}}{\rightleftharpoons}} \underset{\text{ammonio -salt}}{MX} + H.NH_2$$

Ref: Hackh's Dict (1944), 355-R (Franklin E.C.) & 46-L (Ammonia system)

Franklin Institute (FI). An American scientific establishment founded in 1824 in Philadelphia, Pennsylvania and located at 20th & Benjamin Franklin Parkway. Devoted to Science and Mechanical Arts, it has published since 1826 the Journal of the Franklin Institute, while since 1968 it publishes the monthly newsletter "Explosives and Pyrotechnics", edited by Mr Gunther Cohn

The main division of the Institute is the "Franklin Institute Research Laboratory" (FIRL), one of the Amer top ten not-for-profit laboratories. It performs research & development tests and engineering in the basic and applied sciences; also engages in educational activities in the physical and biological sciences. It applies technical expertise to the solution of current problems for government and industry. FIRL employs 250 full-time professional engineers and scientists and handles more than six million dollars in contracts annually. Major departments of FIRL are: Electrical Engineering, Materials and Physical Sciences, Mechanical and Nuclear Engineering, Science Information Services, and Systems Science. In the science of explosives, FIRL has an international reputation for evaluating electroexplosive devices, hazards analysis, and technical information

It should be noted that FIRL's sponsors "Symposiums on Explosives and Pyrotechnics" and that the Eighth Symposium was held in Los Angeles, Calif on Feb 5–7, 1974

Note. Proceedings of past Franklin symposiums summarize the state of the art on

explosives and related topics. Meetings were attended by about 300 representatives from government and industry and each resulted in about 50 papers. Proceedings are available from DDC (Defense Documentation Center, Cameron Station, Alexandria, Va 22314) or NTIS (National Technical Information Service, US Dept of Commerce, Springfield, Va 22151) as follows:

No	Date	Order No	From
	Radiation Hazard Series		
1	May 61	AD-326263	DDC
2	May 63(U)	AD-342306	DDC
	May 63	AD-420595	DDC
	Explosives and Related Topics		
1	Sep 54	AD-066001	DDC
2	Sep 57	AD-153579	DDC
3	Nov 60	AD-323117	DDC
4	Oct 63(U)	AD-350150	DDC
	Oct 63	AD-440764	DDC
5	Jun 67	AD-720454	NTIS
6	Jul 69(U)	AD-514256	DDC
	Jul 69	AD-720455	NTIS
7	Sep 71	AD-742150	NTIS

FIRL also sponsors periodically conducted popular courses on Pyrotechnics and Explosives. Prof J.H. McLain, President of Washington College, Chestertown, Maryland and Mr Gunther Cohn, Senior Staff Engineer of the FIRL, Engr of the (APL) Applied Physics Laboratory are the principal lecturers

The following other activities are part of the Franklin Institute: Library, Science Museum, Planetarium, Journal, Medal Awards, Bartol Research Foundation and Institute Education

Refs: 1) The Franklin Institute Research Laboratories Brochure (Aug 1972)
2) Gunther Cohn of Franklin Institute (Oct 1973)

Franklin's Method of Electric Initiation. It is not generally known but Benjamin Franklin invented electric initiation. He wrote, in a

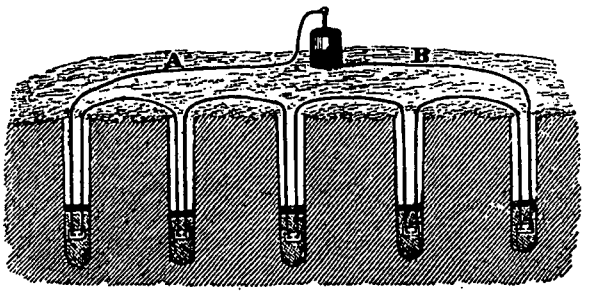


FIG F18

letter to Peter Collinson, "I have not heard that any of your European electricians have ever been able to fire gunpowder by the electric flame. We do it here in this manner. A small cartridge is filled with dry powder, hard rammed, so as to bruise some of the grains; two pointed wires are then thrust in, one at each end, the points approaching each other in the middle of the cartridge till within the distance of half an inch; then, the cartridge being placed in the circuit, when the four jars are discharged, the electric flame leaping from the point of one wire to the point of the other, within the cartridge against the powder, fires it, and the explosion of the powder is at the same instant with the crack of the discharge"
Ref: G. Cohn, Expls&Pyrots **6**(7), 1973

Frantsuzskaya Smes'. Rus for French Mixture, which consisted of PA (Picric Acid) 80 & DNN (Dinitronaphthalene) 20%. It was used for filling some shells, aerial bombs, antitank mines and for demolition charges known as "podryvnyiye shaski" of the same size as those filled with Trotyl (TNT)
Refs: 1) Shilling (1946), p 90 2) Blinov, Vol 1(1938), pp 17–18 3) PATR **2145** (1955), p 6 4) Gorst (1957), p 99
5) Gorst (1972), not listed

Franz's Explosive. It contains Diazodinitrophenol 20, Tetracene 5, Pb ethylenedinitramine 25; Ca silicide 20, Ba nitrate 24, Pb peroxide 6 & gum arabic 0.05% in water as binder. It was proposed for uses in perforating petroleum wells. Its expln temp is

above 300° and it is not too sensitive to impact and friction. Its power is moderate
Refs: 1) A.O. Franz, USP 2708623 (1955) 2) Giua, Trattato **VI** (1) (1959), 405

Frary's Metal. An alloy of lead with small amts of Ba and Ca. It was prepd by electrolytic deposition of Ba & Ca in molten lead. It was as hard as Pb–Sb alloys. Used during WWI for shrapnel bullets
Ref: Hackh's Dict (1944), 355-R

"FRAS". British thickened fuel described under Gel-gas

Fraser & Callaghan patented in Australia at the end of the 19th century Dynamites under the name of **Kallenites**, contg as absorbants dried and pulverized leaves and bark of eucalyptus trees. Some varieties contd saltpeter and NC. Trials conducted in 1899 in quarries of Glebe Island gave excellent results. It was found that these Dynamites do not evolve on expln toxic gases and are, for this reason, particularly suitable for underground work
Ref: Daniel, Dict (1902), 310 & 390

Freeness (or Wetness) and Its Testing. Hackh's Dict (1944), p 357-L defines "freeness" as the extent to which a pulp for the manuf of paper has not been hydrated by beating. Accdg to J.N. Stephenson, Edit, "Preparation and Treatment of Wood Pulp", McGraw-Hill, NY, Vol 1 (1950), pp 943–56, the "freeness" (slowness, or wetness) is the quality of pulp stock that determines the rate at which it parts with water when being formed into a sheet on a wire screen or perforated plate. The freeness test gives information regarding the drainage behavior to be expected on the paper machine, and it also gives some indication of the strength, bulk and finish of the final paper

The following freeness testers are described in the above book:

a) Canadian Standard Freeness Tester (pp 942–951);

b) Schopper-Riegler Freeness (Slowness) Tester (pp 951–56)

c) Williams Precision Freeness Tester (pp 955–56)

d) Drainage Tester (pp 956–57)

e) British Drainage Tester (pp 957–60)

According to L. Sheldon, PB Rept **12662** (1945) the Germans used the Schopper-Riegler Device for testing the crepe paper used for the manufacture of nitrocellulose

Free Radical and Free Radical Propellants. Accdg to Hackh's (Ref 1), a *free radical* is an org compd in which all the valences are not satisfied; hence, an unsaturated molecule, as:

$2Ph_3C \rightleftharpoons Ph_3C - CPh_3$

triphenylmethyl from hexaphenylethane;

$2Ph_2N \rightleftharpoons Ph_2N - NPh_2$

diphenylnitrogen from tetraphenylhydrazine

In a strict sense also:

$2NO_2 \rightleftharpoons N_2O_4$

nitrogen peroxide from dinitrogen tetroxide

Free radicals may also be defined as "unstable fragments of organic compounds". Small free radicals exist in flames and in many high-speed chem reactions. Generally they last for only millionths of a second before combining violently with each other

Free radicals are of particular interest in rocket propulsion, since their presence in chemical fuels might give rockets much higher thrust (thru the increased momentum discharge) than do conventional chem fuels, if economical ways can be found to generate and use them (Ref 3)

Accdg to Boehm (Ref 2), the upper reaches of the atmosphere where matter is extremely sparse, are relatively rich in free radicals; in that region, chiefly atomic forms of hydrogen, nitrogen and oxygen are present. Conceivably, a ramjet satellite flying a hundred miles or more above the earth could scoop up free radicals and use them as fuel. A scoop 30-feet in diam could collect enough radicals to generate about 1 watt of power – enough perhaps to effect the drag in the atmosphere and keep a satellite in its orbit indefinitely

It may be possible to stabilize the free radicals at sea-level and feed them directly into rocket engine. In 1954, two scientists at the Natl Bur of Standards Labs sent a stream of nitrogen thru an electric discharge and immediately froze it at −452°F. A small fraction of a percent of the frozen material was atomic nitrogen as free radical. The potential propulsive power of free radicals is astonishing. A fuel consisting of 10% H and 90% H_2 would be more powerful than any known liquid chemical propellant; and pure H atoms reacting with each other to form H_2 would theoretically produce a specific impulse of 1800 – twice that of nuclear rocket engine

Refs: 1) Hackh's Dict (1944), 357-L 2) G.A. W. Boehm, Fortune, Dec 1957, pp 172 & 177 3) Anon, "Elements of Armament Engineering", Part 1, "Sources of Energy", ORDP **20-106**(1960), p 3-27

Free Rocket. A rocket having fixed fins, but no control surface, that is, no provision for guidance

Ref: Glossary of Ordn (1959), 123-L

Free Run. As applied to guns, the travel of a projectile from its original position in the gun chamber until it engages with the rifling of the bore

Ref: Glossary of Ordn (1959), 123-R

Free Surface. See Vol 4, p D349-L

Free Surface Velocity Method for Determination of Detonation Pressure. See Vol 4, pp D487-R to D488-L, under Detonation (and Explosion), Pressures and Their Measurements

Free Volume Theory of the Liquid State Developed by Eyring et al and by Lennard-Jones Devonshire. See Vol 4, pp D349-L & R

Free Volume Theory of Lennard-Jones-Devonshire (LID). See under Detonation, Free Volume Theory of the Liquid State Developed by Eyring et al and by Lennard-Jones-Devonshire in Vol 4, p D349-L & R

Free Volume Theory of Multicomponent Fluid Mixtures. See "Detonation, Free Volume Theory in Multicomponent Mixtures" in Vol 4, p D349-R and in the paper of Z.W. Salsburg & J.G. Kirkwood, JChemPhys **20**, 1538–43(1952)

Freezing of Dynamites and Antifreezes. See under DYNAMITE in Vol 5, pp D1588-R to D1593-L

Freezing (or Frigorific) Mixture or Cooling Bath. A mixture of substances that absorbs heat and thus lowers its temperature or that of its immediate surroundings. Used in analytical and synthetic chemistry for reactions at low temperatures. Following are aqueous mixts listed in Ref 1:

a) 1 pt NH_4NO_3+1 pt Na_2CO_3+1 pt H_2O lowers to −20°C

b) 5 pts NH_4Cl+10 pts powdered ice or snow gives −5° to −18°

c) 5 pts NaCl+5 pts NH_4NO_3+12 pts ice or snow gives −15° to −25°

d) 3 pts $CaCl_2$+1 pt ice or snow gives −40° to −70°

e) 10 pts dil H_2SO_4+8 pts ice or snow gives −65° to −90°

A more complete list of aqueous freezing mixtures is given in Ref 2

The following non-aqueous cooling baths are listed in Ref 2:

a) Alcohol–carbon dioxide gives −72°

b) Ammonia, boiling point −33°

c) Chloroform–carbon dioxide gives −77°

d) Ether–carbon dioxide gives ca −78°

e) Liquid air, bp −190°

f) Liquid nitrogen, bp −196°

g) Liquid oxygen, bp −183°

Refs: 1) Hackh's Dict (1944), 357-R 2) Lange's Hdb (1961), 1191

Freezing (Solidification, Congelation or Setting) Point (fr p) and Its Determination. Freezing point is the temperature at which a liquid or solution solidifies. It is the temperature at which the liquid and solid states of a substance are in equilibrium at a given pressure (usually atmospheric). The second definition may be applied to *Melting (or Freezing) Point*, which may also be defined as the temperature at which a solid changes to a liquid. For pure crystalline substances, mp is usually identical with fr p, while for many mixtures, they are not identical. For example, fats and waxes do not solidify until they have been cooled several degrees below their mp's. If a liquid be cooled under pressure the fr p rises if the solid is of higher specific gravity than the liquid; if it is of lower specific gravity, as in the case of water, the fr p is lowered. Since the fr p varies slightly with pressure, the pressure should be stated in reporting a freezing point. However, atmospheric pressure is commonly understood unless otherwise stated

The simplest method for freezing (setting) point determination is one employing an evaporating dish. This method was commonly used in US Ordnance Works during WWII for rapid detn of setting point of TNT. It is described in Ref 1, Chap V, p 3. In the laboratory of Keystone OW, Meadville, Pa, the dish was replaced by Pyrex custard cup because it was more convenient to handle. The cup procedure described in Ref 2 and in Vol 2, of Encycl, p C7-L was used for calibration of thermometers by using liquids of known freezing points, or for detn of frp's of unknown liquids, using calibrated thermometers. Another simple method employed a Pyrex cylinder, thermometer, and a stirrer with a ring surrounding the thermometer. This method is described in Vol 2, p C6-R. More complicated apparatus is shown in Fig on p A613-L of Vol 1. It has been used in Sodium Azide Laboratory but can be used for many other compds. Finally, the best and more precise apparatus is that shown in Fig on p D1473-R of Vol 5. It has been used for determination of setting point of diphenylphthalate. It is a copy of apparatus described in Ref 1, Chap V, p 5, which is a modified version of US Joint Army-Navy Specification JAN-T-248 for TNT (Compare with Melting Point)

Refs: 1) Clift & Fedoroff, Vol 1(1943), Chap V, pp 3 to 5 (Evaporating dish method and modified Spec JAN-T-248 Method) 2) Ibid, Vol 3(1944), Chap V, pp 4–6 (Custard cup method) 3) The Van Nostrand Chemist's Dictionary (1953), pp 304-R & 305-L 4) J.A. Richards, F.W. Sears, "Modern University Physics", Addison-Wesley Pubg Co, Reading, Mass (1960) 5) Lange's Handbook of Chemistry (1961), 1733-R

Freezing Tests for Dynamites. See Vol 1, p XIII and under DYNAMITE in Vol 5, pp D1588-R to D1593-L

More detailed description is given in USBurMinesBull 346 (1931), 27–9

Freiberg (Poudre de mine de). A French mining explosive, claimed to be economical to use: Na nitrate 61.65, sulfur 17.25 & charcoal 17.35%

Ref: Daniel (1902), 310

Freins de bouche. Fr for Muzzle brakes

Freins de tir. Fr for Recoil brakes

French Ammonals S and C. See Vol 1, p A290 (Table), under AMMONAL

French Ammonium Nitrate Gelatin Dynamites. See Vol 1, p A368 (Table), under AMMONIUM NITRATE GELATIN

French Ammunition and Explosives of WWII

The principal types were *HE Service Bombs*, which were made of hardened steel, forged or

Table F14

Designation and Type	Overall Length	Max. Diameter	Type of Filling	Wt of Filling	Total Wt	Fuze
14kg AP (Naval)	–	3¾	PA&TNT	1¾ lb	31 lb	–
73kg Type G2, Antisubmarine	–	8.66	TNT	106	160	Nose No 1 Tail No 8
150kg Type 12, Antisubmarine	–	14	TNT	224	330	Same as above
224kg Type K (Naval)	–	14.75	PA or PA+TNT	240	490	Nose No 10 Tail No 8
222kg Model 1938 AP (Naval)	–	10¾	PA+TNT	50	500	Tail No 7
410kg Type L-AP (Naval)	–	15¾	PA or PA+TNT	265	900	Nose No 9 Tail No 8
438kg Model 1938 AP (Naval)	–	11.9	PA	24	1000	Tail No 7
720kg Type M (Naval)	–	20.5	TNT	860	1600	Nose No 11 Tail No 8
125kg GP-HE (Naval)	61.5''	12.2	TNT	Unknown	12.5	Nose only
10kg (PA) Antipersonnel Type I and Type II	21.5	3.54	MMn	2.5	21.0	Nose only
50kg (A) GP-HE	39.0	6.15	MDn	22.0	88.0	Nose Type H Tail–3B15
50kg (GA MMn) GP-HE	46.5	7.85	MMn	63.0	112.0	Tail M No 2
50kg (DT No 1) GP-HE	46.5	8.25	MDn or DD 60/40	44.0	123.0	Tail 3B15
50kg (DT No 2) GP-HE	46.5	7.7	Same	42.0	123.0	Same
50kg (DT No 3) GP-HE	46.5	7.86	Same	44.0	123.0	Same
100kg GP-HE	56.0	10.8	Same	258	Unknown	Nose & Tail
200kg GP-HE	62.0	14.5	Same	503	Unknown	Nose & Tail
500kg Model 1930 GP-HE	84.0	19.3	Same	600	1285	Nose & Tail
500kg No 1 GP-HE	83.0	21.4	Same	662	1182	Same
500kg No 2 GP-HE	79.0	19.6	Same	595	1148	Same
40kg Fragmentation Bomb	37.75	6.25	Unknown	Unknown	Unknown	Nose No 3 Tail, unknown
1 kg Incendiary Type A	14.5	Unknown	Thermite	Unknown	1 kg	Nose only
10kg (Model 1927) Incendiary	22.1	3.54	Mg Incendiary	Unknown	8 kg	Nose only
Smoke Bomb	9.45	2.75	Titanium Tetrachloride	100ml	Unknown	None

drawn. The service HE bombs of the old pattern were painted grey, while the new pattern were painted yellow. The *Incendiary Service Bombs* of the new pattern have a red body and a green tail, and the *Smoke Service Bombs*, a black nose, green body and tail. There were also Practice Bombs, Instruction Bombs and Ballast Bombs. The hardened steel bombs were usually filled with MMn, the steel bombs with MDn and other bombs with DD 60/40. Most of the Naval HE bombs were filled with Mélinite, Mélinite plus Tolite or Tolite alone. The incendiary bombs usually contained thermite or magnesium incendiary mixture, and some smoke bombs contained a titanium tetrachloride mixture

Table F14 lists bombs described in Ref, pp 177–87

Abbrs: **AP** – Armor-piercing; **DD 60/40** – PA/DNPhenol; **DNN** – Dinitronaphthalene; **GP** – General Purpose; **HE** – High Explosive; **MDn** – DNN/PA–20/80%; **Mélinite–(PA)** – Picric Acid; **MMn** – MNN/PA–30/70%; **MNN** – Mononitronaphthalene; **Tolite** – (TNT)–Trinitrotoluene

Note: French weapons of WWII are listed in Table F19

Refs: 1) Anon, "Italian and French Explosive Ordnance", OP **1668**(1946), pp 177–87 2) Anon, "Italian and French Explosive Ordnance", **TM9–1985–6/TO 39B–1A–8**(1953), 177ff

French Anarchist's Explosive, known as **Green Powder,** used in bombs of revolutionists during 1892's criminal assaults, contd: K chlorate 49, K ferrocyanate 28 & sugar 23%. This expl mixt, belonging to Cheddite-class, was very sensitive and dangerous to handle
Refs: 1) Pascal (1930), 218 2) Clift & Fedoroff, Vol I(1943), p G5

French Armor. After WWII, France concentrated on light armored vehicles and did not try to design and develop a complete armored system. Battle tank regiments used American Patton tanks. Since 1950, stress was laid on mobility and a light air transportable tank was designed. This led to development of the AMX 13-ton tank. Present inventory includes personnel carriers, command vehicles, self-propelled howitzers, special purpose engineer vehicles and recovery vehicles. The following paragraphs describe basic features of this inventory:

AMX 13-ton Tank. Tank's loaded weight is 14½ metric tons, road speed 37mph. Its range is 150 miles or 13 hours. Tank is 7 feet, 6 inches in height and its main weapon is a 75mm gun with automatic reloading mechanism. Tank fires HE armor-piercing shells with muzzle velocity of 3280fps. A 7.5mm coaxial machine gun and four smoke-shell projectors complete the armament

AMX-SS-II Tank. Some AMX tanks have been equipped with four SS-II antitank missiles mounted in fire position in front of turret. Missiles are manufd by NORD Aviation, weigh 62.7 lbs and carry a shaped charge. Maximum range is 11480 ft and can be reached in 22 sec. Missiles are wire guided

AMX Personnel Carrier Vehicle weighs 13½ tons and has same technical features and performance as its parent tank. It can carry 12 men plus a driver. Onboard armament consists of 7.5mm machine gun in a 360° turret. It is convertible to a command vehicle, ambulance, or cargo vehicle with 3-ton carrying capacity

AMX 105mm Self-propelled Howitzer. Built on AMX chassis and has same performance. Its range is 9 miles. It is also equipped with anti-aircraft machine gun and a machine gun for close defense. It weighs 16 tons combat loaded with a 5-man crew and fifty-four 105mm rounds on board

155mm Self-propelled Howitzer. Mounted on a self-propelled AMX carriage, this howitzer has a range of 12.4 miles, loaded weight is 17 metric tons

30mm Self-propelled Double-Barreled Gun. An AMX chassis carries a turret equipped with two 30mm guns which fire HE or armor-piercing projectiles at a muzzle velocity of 3280fps. Max range 8200 ft and rate of fire

is 20 rounds/sec. Also has "Black-Eye" surveillance radar. Vehicle weighs 16 metric tons combat loaded with crew of a driver and two turret gunners

AMX Recovery Vehicle. Same technical features as in AMX tank, weighs 14½ metric tons; has 3 man crew, a generator, winch with a tractive power of 15 tons and a crane to lift 5 metric tons

Engineer Combat Vehicle. Can transport an engineer squad and its equipment. Used to clear roads and lay down various obstacles. Crew consists of a driver, machine gunner, a non-commissioned officer and seven soldiers. Has a mechanical shovel, winch and a crane with a 5 ton lifting capability. Armament is a 12.7mm machine gun. A 3-ton trailer carries all materials needed for its mission

Light Bridge-Laying Tank. Built on AMX chassis, can transport and lay a bridge of 25 ton class. Has a 3-man crew

Other armored vehicles include: the Armored Reconnaissance Vehicle (EBR), Light Armored Combat Car and the new AMX 30-Ton Tank

Ref: Service d'Information, d'Études et de Cinematographie, Ministère des Armées, France, Ordn **50**, No 273, pp 291–95 (1965)

French Commercial Explosives of Non-Permissible Type. See Vol **3**, p C438. Included as of 1966 were: Explosifs antigrisouteux; Explosifs chloratés ou Explosifs du Type OC, also known as Cheddites; Explosifs nitratés ou Explosifs du type N; Explosifs nitratés à la pentolite; Explosifs nitratés à l'aluminium; Explosifs nitratés résistant à l'eau; Explosifs plastiques; Sevranite n° 1, Sevranite n° 2; Sofranex A and Tolamite

French Commercial Explosives of Permissible type. See Vol **3**, pp C450-R & C451-L. Their general name is *Explosifs antigrisouteux* and they included as of 1966: Grisou-dynamite chlorurée n° 1, Explosif du type N, n° 7; Explosif du type N, n° 9, Grisou-dynamite chlorurée n° 14 (1948); Grisou-dynamite chlorurée n° 15 (1949); Explosif du type N, n° 62; Explosif 123–CSE (Commission des Substances Explosives) – 1948; Explosif 136–CSE–1948 and Explosif 68–CSE–1949

French Compound. A substance of unknown composition obtained when dilute nitric acid reacts with a mixture of TNT and lead (or iron). French Compound easily ignites when brought into contact with nitric acid vapor at 100°. Either fragmentation or HE bombs can be loaded with this material and the method of loading is either lump or by compression of powdery material. This material was used by Russions

(Compare with French Mixture)

Ref: Aberdeen Proving Ground, "Allied and Enemy Explosives" (1946)

French Cyclonite, known as Hexogène. See Vol **3**, p C615-L, under CYCLOTRIMETHYLENETRINITRAMINE

French Cyclonite Binary and Ternary Mixtures. See Vol **3**, p C626-R. Included as of 1966 were the following: Hexogène/cire d'abeille (RDX & beeswax), Hexolite or HT (Hexogène–Tolite) (RDX–TNT); Sevranite n° 1; Explosif plastique PE1; Explosif plastique PE3A; Explosif plastique TG; and Explosif plastique TN

French Demolition Explosives. See Vol **3**, pp D58-R & D59-L, under Demolition Explosives

FRENCH DYNAMITES. Accdg to Pascal (Ref 1), Vennin, Burlot & Lécorché (Ref 2), and Pepin Lehalleur (Ref 3), the French Dynamites of before WWII were divided into:

A. Dynamites à absorbant inerte, which are subdivided into *Dynamite n° 1* – NG 75 & kieselguhr 25%; *Dynamite n° 2* – NG 35 &

kieselguhr 65% and *Dynamite n° 3* – NG 25 & kieselguhr 75% (Ref 2, p 547)

B. Dynamites à absorbant active *(pulvérulentes)*, as for example, *Dynamite n° 3* of the Société Génerale pour la Fabrication de la Dynamite, which contained NG 22, saltpeter 66 & charcoal 12% (Ref 2, p 551)

C. Dynamites à gélatinisant active. They employ as gelatinizer for NG, the "coton azotique n° 2" (CA^2) which is NC of ca 12% N, known in US and Gt Britain as Collodion Cotton (abbr CC) and in Germany as Kollodiumwolle

French Gelatin Dynamites can be subdivided into the following categories (Ref 2, p 553)

Ca) **Dynamites-gommes.** They contained large amounts of NG, and small amounts of CC. Those contg 70 to 83% NG and small amt of CC contained other ingredients, as can be seen in Table F15

Cb) **Dynamites gélatinées.** They contained smaller amounts of NG, slightly smaller amounts of CC, but larger amount of K nitrate. Two formulations are listed in Table F16

Table F15

Components	Designation			
	Gomme A	Gomme B	Gomme C	Gomme E
NG	92–93	83	70	49
CC	8–7	5	4	2
K nitrate	–	9	16	36
Cellulose	–	3	–	–
Wheat flour	–	–	8	3
Woodmeal	–	–	2	10

Note 1: Accdg to Davis (Ref 4, p 346), Gomme A was also known as *Dynamite-gomme-extra-forte* and Gomme B as *Dynamite-gomme-potasse;* if K nitrate was replaced by Na nitrate the name became Dynamite-gomme-soude

Note 2: Compn of low-freezing Gomme B is given in Table 1, in Vol **5**, p D1591, under DYNAMITE, Historical

Note 3: Compn of Gomme E is taken from Ref 4, p 346

Table F16

Components	Designation	
	Gélatinée n° 1	Gelignite A
NG	57	65
CC	3	3.8
K nitrate	34	23.5
Woodmeal	6	–
Cellulose	–	7.5
Ocher	–	0.2

Davis (Ref 4, p 346) gives among the French Gelatin Dynamites, the compns listed here in Table F17

Table F17

Components	Designation			
	Gélatine A	Gélatine B (potasse)	Gélatine B (soude)	Gélignite
NG	64	57.5	57	58
CC	3	2.5	3	2
K nitrate	–	32.0	–	28
Na nitrate	24	–	34	–
Woodmeal	8	8.0	6	9
Flour	–	–	–	3
Mg carbonate	1	–	–	–

Note: Fr low-freezing Gélatine is listed in Table 1, under DYNAMITE, Vol 5, p D1591

Pepin Lehalleur (Ref 3) lists on p 334 the *Gomme BAM* which is one of the permissible Gelatins: NG 60, CC 3, AN 31 & cellulose 6% and on p 335 he lists a non permissible Gelatin which contains NG 57, CC 3, alkali nitrate 34, woodmeal 4, flour 1.8 & ocher 0.2%

Pepin Lehalleur also lists the following *Gommes incongélables* (Nonfreezing Gelatins): 1) Manufd by the Nobel Française: NG 75, CC 5, alkali nitrate 10, DNT 8 & woodmeal 2%; and 2) Manufd by the Société d'Explosifs et de Produits Chimiques: NG 66.4, NGc 16.6, CC 5, alkali nitrate 10 & woodmeal 2% (Ref 3, p 336)

Note: Vennin et al (Ref 2, p 557) use the term "antigel" instead of "incongélable"

Two low-freezing *Vergé Dynamites* are listed in Table 1, under DYNAMITE in Vol 5, p D1591

Cc) **Dynamites à faible taux de nitroglycérine qui se présent sous forme pulvérulente.** To this group belong powdery Dynamites with NG content below 30% and CC content 0.5–1.0%. Those which are "permissible" are known as "Explosifs antigrisouteux", "Grisounites" and "Grisoutines". The meanings of these terms as well as of Belgian term "Explosifs SGP" are explained in Vol 1 of Encycl, p A466-R

Vennin, Burlot & Lécorché (Ref 2), listed on p 557, under the name *Grisou-dynamite-couche-salpêtrée (antigel)*, the following compn: NG 9.6, NGc 2.4, CC 0.5, AN 82.5 & K nitrate 5%. A similar compn, but without NGc, is listed by them in Table on p 554, together with Grisou-dynamite-couche and Grisou-dynamite-roche. These three compns were listed in Vol 1 (Ref 7), Table on p A467-L, entitled "Antigrisou (Explosifs)"

Vennin et al also listed on p 554 as permissibles: *Lignamite-couche (or Gricellite-couche), Martinite* and *Tolamite*. Tolamite was described in Vol 3 of Encycl (Ref 7) on p C437-R, under nonpermissibles, while Lignamite and Martinite will be listed here under L & M

Davis (Ref 4) listed in Table on p 351, four "Grisou-dynamites" of the same compns as given by Vennin et al (Ref 2, pp 554 & 557), of which one compn, *Grisou-dynamite-roche-salpêtrée:* NG 29.0, CC 1.0, AN 65.0 & K nitrate 5%, was not listed in Vol 1, p A467-L

One of the Fr low-freezing *Grisou-dynamites* is listed in Table 1, under DYNAMITE, Historical in Vol 5, p D1591

Some "antigrisou" expls contained no NG. A brief list of such "substitute" Dynamites is given in Vol 1, p A466-R (Ref 7)

Pepin Lehalleur (Ref 3) lists on p 342 three Dynamites manufd at Cugny. These expls are listed in Vol 1 of Encycl, p A356, under the names of French 1, 2 & 3. Pepin Lehalleur also lists a similar Dynamite manufd by the Société Nobel. This expl will be described in this section under *Nobelites*

The following "types réglamentaires en France pour les travaux dans les mines grisouteuses" (before WWII) were listed by Pepin Lehalleur (Ref 3, p 343) (See Table F18)

Table F18

Components	Designation			
	Grisou-dynamite-Chlorurée nº 1	Antigrisou		Antigrisou Couche Salpetrée
		Roche	Couche	
NG	16.0	29.1	11.7	12.0
NGc	4.0	–	–	–
CC	0.5	0.9	0.3	0.5
K nitrate	–	–	–	5.0
AN	55.0	70.0	88.0	82.5
Cellulose	2.5	–	–	–
Na chloride	22.0	–	–	

Vivas, Feigenspan & Ladreda (Ref 5), listed on pp 390, 391, 395 & 396 several French Dynamites of practically the same compn as those given in French books listed here as Refs 1, 2 & 3

The following "nonpermissible" Dynamites are listed in Vol 3 of Encycl (Ref 8, p C438). They are described by Médard (Ref 6, p 209–19). He subdivided them as: *Explosifs chloratés*

ou Explosifs du type OC, Explosifs nitratés à l'aluminium; Explosifs nitratés résistant a l'eau; Explosifs plastiques; Sevranite No 1; Sevranite No 2; Sofranex A; and Tolamite

The following "permissible" Dynamites are listed in Ref 8, pp C450 & C451, accdg to classification of Médard (Ref 6, pp 219–22): *Grisou-dynamite chlorurée n° 1* (1932); *Explosif du type N, n° 7* (of Burlot & Schwob); *Explosif du type N n° 9* (of Audibert) (1938); *Grisou-dynamite chlorurée n° 14* (1948); *Grisou-dynamite chlorurée n° 15* (1949); *Explosif du type N n° 62* (of Médard); *CSE* (Commission des Substances Explosives) Explosifs, such as *Explosif 123–CSE–1948; Explosif 136–CSE–1948;* and *Explosif 68–CSE–1949*

Refs: 1) Pascal (1930), pp 221–24 2) Vennin, Burlot & Lécorché (1932), 547–57 3) Pepin Lehalleur (1935), 334–343 4) Davis (1943), 350–51 5) Vivas, Feigenspan & Ladreda, Vol **2** (1946), 390–91 6) L. Médard, MP **32**, 209–24 (1950) (Recent progress and actual tendencies in the field of mining explosives in France) 7) Fedoroff & Sheffield, "Encyclopedia of Explosives", PATR **2700**, Vol **1** (1960), pp A466-R & A467-L 8) Ibid, Vol **3** (1966), pp C438 & C450-R to C451-L

FRENCH DYNAMITES AND THEIR SUBSTITUTES.

Following expl compns, many of them obsolete, are described in Vols **1, 2, 3** and in Vol **5** under DYNAMITES AND THEIR SUBSTITUTES: Aluminum Containing Dynamites, Amide ou Explosif amylacé, Ammonique Dynamite, Ammonite, Anagon, Antigrisou n° 1, n° 2 & n° 3; Antigrisou II, Antigrisou-couche, Antigrisou-couche salpêtrée, Antigrisou-roche, Antigrisouteux explosifs N n° 7, N n° 9 & N n° 62 (See Vol **1**, p A466 & A467), Antigrisoutines, Bayon, Bender, Bengaline, Berclavite, Bergé, Berthelot, Berthollet, Blanche dynamite de Diller, Blanche dynamite de Paulilles, Boritines de Turpin, Borlinetto, "C" (Explosifs), Carbite, Carboazotine, Carbonites (French Modifications), Carrière, Casthelaz et Désignolle, Castro, Catactine, Cauvel et Baron, Cellamite, Charbonneux, Chauvet, Cheddites, Colombia (or Columbia), Cornil ou Poudre blanche, Cotter, Crésylites, CSE (Explosifs), Cugnite, "DD" (Explosif de), DeCastro, Defraiteur, Delattre, Delhorbe, Désignolle, Delhorbe, Domergue, Duplexite de Turpin, Dynamite gélatinée, Dynamite-grisoutine, Dynamite "O", Dynamite de Vonges, Dynamogène, Dynamoite and Dynastite Explosifs antigrisouteux, Explosif "N", Explosifs "N", "NDNT", "NO", "N4", "NT", "NTN", "N2TN" and "NX"; Explosif "O", Explosif "93" (or Prométhée), Explosif "P", Explosif "S", Explosif "Sc", Explosifs de sûreté, Explosif type C, Explosif type N, Fahrm, Favier, Fenices, Fielder, Fluorine, Fontaine, Forcites, Fortex, Gélatinée n° 1 (See Table 11), Gélatines A & B (See Table 12), Gélignites (See Tables 11 & 12), Gommes A, B & C (See Table 10), Gomme BAM (See under Table 12), Gomme E (See Table 12), Gommes incongélables (See under Gomme BAM); Grisou-dynamite-couche, Grisou-dynamite-couche-salpêtrée and Grisou-dynamite-roche are described in Vol **1**, p A467-R as Antigrisou (Explosifs)

The following French Dynamites are described, together with expls of other nations, in this list under the letters G, etc: Grisou-naphthalite-couche, Grisou-naphthalite-roche

French Explosive Industry of After WWII was reviewed by LeRoux in Revue de l'Industrie Minerale **33**, 276–84 (1952)

French Explosives. See under Explosifs in this Vol and under individual names, such as: Anilite, Cheddite, Favier explosifs, Grisou-dynamites, Hexogène, Mélinite (PA), Nitramidon (NS), Poudre noire (BkPdr), Schneiderite, Tolite or Trotyl (TNT) and others

French Gap Test. See under Coefficient de self-excitation (CSE) in Vol **3**, p C390-L and under French Tests in this Section

French Gelatinized Explosives (called Gomme, Dynamite-gomme, Gélatine). Expls resembling in their compositions American Blasting Gelatin (called in France, Dynamite-gomme-extra forte), Gelatin Dynamites or British Gelignites
Refs: 1) Marshall **1**(1917), 372 2) Encycl, Vol **5**(1972), Table 1, p D1591

French Grisou-Dynamite. See under DYNAMITE in Vol **5**, p D1591, Table 1, last column

French Ground-to-Ground Strategic Ballistic Missile, abbr as **SSBS** (Sol-Sol-Ballistique-Stratégique) is an ICBM type weapon described in Ref
Ref: Anon, Ordn **48**, 490(1964)

French Lead Block (or Trauzl) Test. See Coefficient d'utilization pratique (CUP) in Vol **3** and on pp IX–X in Vol **1** of Encycl
Addnl Ref: L. Médard, MAF **33**, 337–388(1959)

French Mixture (Frantsuzskaya Smes', in Russian). Mixture of PA (Picric Acid) 80 & DNN (Dinitronaphthalene) 20%, which may be considered as phlegmatized PA (Refs 1, 3, 4 & 5). It was used both in France and in Russia as a bursting charge in mines, bombs and shells. A slightly less sensitive and less powerful mixture was *Russkaya Smes'* (Russian Mixture)(Ref 4), called *Russkii Splav* (Russian Splav) in Refs 1 & 3. It consisted of PA 51.5 & DNN 48.5%. The name *Russkaya Smes'* was given by Blinov (Ref 2) to a mixture of AN 50, TNT 38 & TNX 12%. French Mixture was used by the Russians for loading medium caliber shells (larger than 76.2mm) as late as WWII
Refs: 1) Shilling (1946), 102 (Frantsuzskaya Smes'); 90 (Russkii Splav) 2) Blinov, Vol **2**(1949), 64–5(Russkaya Smes') 3) Fedoroff et al, PATR **2145**(1955), p Rus 6-R(Frantsuzskaya Smes'); p Rus 20-R(Russkii Splav) 4) Gorst (1957), 99(Frantsuzskaya Smes'); 99(Russkaya Smes') 5) Gorst(1972), 136 (Mixture of PA 80 & DNN 20%, w/o naming it Frantsuzskaya Smes')

French Low-Freezing Dynamites are expls having compns similar to Amer low-freezing Dynamites, such as for example: NG 66.4, EGDN 16.6, Collod Cotton 5.0, K nitrate 10.0 & woodmeal 2.0% (Ref 1). Two Fr low-freezing gelatins are listed in Ref 2
Refs: 1) Marshall **1**(1917), 373 2) Encycl of Explosives, PATR **2700**, Vol **5**(1972), p D1591, Table 1

French Military Explosives, Requirements. Accdg to Pepin Lehalleur (Ref 1, pp 53–6) the following requirements were specified for **explosifs militaires:**
1) Great power and brisance in relation to volume of explosive, which means great density of loading
2) True and complete detonation by an initiator even at atmospheric pressure and without tamping
3) Insensibility to shock on discharge of expl loaded in a projectile and insensibility to impact of a bullet
4) Inalterability by heat, moisture and contact with the walls of container (shell). Inoxibility by air or on contact with oxidizers
5) Constance of properties and absence of exudation
6) For expls used by sappers in military mines, no poisonous gases, such as CO, should be produced on detonation
7) Facility of producing explosives from domestic materials and domestic factories

The following explosives were used before WWII by the French for military purposes: Trotyl (TNT), Mélinite (Trinitrophenol or Picric Acid), Dynamite gomme (NG/CP_2–92/8), Penthrite (PETN or Pentaerythritol Tetranitrate) and Hexogène (RDX or Cyclonite). Their properties are listed in Table, p 54 of Ref 1

Explosives used by the French during WWII are given in Ref 2
Refs: 1) Pepin Lehalleur (1935), pp 53–56
2) Anon, "Italian and French Explosive Ord-

nance", **TM9/1985-6/TO 39B-1A-8** (March 1953), pp 177ff (Bombs, Fuzes, Mines & Igniters)

French Safety Explosives *(Explosifs de sûreté).* Strictly speaking, this term should be confined to *explosives safe to handle and to transport* (corresponding to German term "Handhabungssichere"), but sometimes the term was used in regard to explosives "safe to use in gaseous coal mines". The latter explosives are known now as *explosifs antigrisouteux,* which correspond to German "Schlagwettersichere", Permissible Explosives" in USA) or "Permitted Explosives" in Great Britain (Ref 1, p 582 & Ref 4, p C450-R)

Accdg to Vennin, Burlot & Lécorché (Ref 2, p 212) the main requirement for "explosifs de sûreté" is insensitivity to friction and only low (if any) sensitivity to shock

From the point of safety in transportation, Pepin Lehalleur (Ref 3, pp 495–97) divides expls, proplnts and pyrotechnics into the following four groups, starting with the more dangerous materials:

1) Most dangerous group: Black Powder and colloidal propellants, in bulk placed in boxes, and chlorate explosives, not cartridged. Dynamites: with attached caps; fuses, detonators, nonelectric & electric and Nitrocelluloses wetted with at least 25% water. Aromatic nitrocompounds, packed in double containers: the inner ones from leather, cardboard, heavy paper envelope, while the outer ones of wood or copper. The fulminates must be transported in vessels filled with water

The boxes must be transported in special, covered vehicles

2) Second group, less dangerous to transport than the first one, comprises: pyrotechnic devices, detonating cords w/o caps attached, and Mélinite (PA) cartridges, all placed in barrels of small resistance

3) Third group comprises the "explosifs de sûreté" (to which belong most AN expls) packed in carbon or wooden boxes; then NC wetted with more than 25% water, placed in waterproof containers and finally railroad torpedoes placed in special containers

4) Fourth group, less dangerous to handle and transport, comprises: shotgun cartridges (for sporting), and military cartridges in finished state and properly packed; fuses without caps attached and igniters for fuses – all these materials must be packed either in wooden or in tinned iron boxes

The materials of the first three groups must be loaded and unloaded in day-time

The above regulations are those of pre-WWII, but we have no info about current regulations

Refs: 1) Marshall **2**(1917), 582 2) Vennin, Burlot & Lécorché (1932), 212 3) Pepin Lehalleur (1935), 495–97 4) Fedoroff & Sheffield, PATR **2700**, Vol **3**(1966), p C450-R

FRENCH TESTS FOR EXPLOSIVES AND PROPELLANTS (Arranged in alphabetical order under French names)

Brisance par l'appareil de Calvet. Se Ref 12

Brisance par épreuve de Chalon (Brisance by Chalon Test). See Ref 21, p B299-R & Ref 22, p C493-L

Coefficient de self-excitation (Coefficient of Self-excitation Test), abbr CSE; Aptitude à transmettre la détonation à distance (Capability to Transmit the Detonation at a Distance); Détonation par influence (Detonation by Influence) or Détonation sympatique (Sympathetic Detonation). See Ref 7, p 799; Ref 17, p 74; Ref 19, p 342; Ref 20, p 10 and Ref 23, pp D395-R to D405-L

Coefficient d'utilisation pratique, abbr as CUP or cup (Coefficient of Practical Utilization) or Épreuve de travail spécifique (Specific Work Test). It is Dautriche's modification of Trauzl Lead Block Test. See Ref 20, pp IX & X; Ref 4, p 472; Ref 8, p 171; Ref 10, p 66 & Ref 19, p 344. Also in Ref 24, p E10-R, as Travail spécifique rélatif

Combustion en gouttière de 20mm (Combustion in a 20mm Trough), also called Épreuve de propagation dans une gouttière de 20mm de diamètre (Propagation Test in a Trough of 20mm Diameter). See Ref 24, p E138-L

Combustion en tas conique (Combustion in a Conical Pile) or Aptitude à l'inflammation, essai (Sensitivity to Inflammation Test in Conical Pile) (See Ref 24, pp E138-R & E139-L)

Courbe de compressibilité des explosifs solides (Curve of Compressibility of Solid Explosives). See Ref 24, p E139-R

CUP or cup. Abbr for Coefficient d'utilisation pratique

Dautriche, Méthode de (pour mesurer la vitesse de détonation) (Dautriche's Method for Measurement of Velocity of Detonation). See Ref 24, p E140-R which includes 8 refs. Detailed description is given in Ref 23, pp C311-R (with Fig) and p C312-L

Densité, Épreuves de (Density Tests). See Ref 17a, p 198; Ref 22, pp D64-R to D81-R

Densité de tassement, Essai (Packing Density Test). See Ref 24, p E137-L

Desmaroux, Épreuve de (Desmaroux Test). See Ref 5, p 24; Ref 11, p 90 & Ref 22, p D90-R

Détonation par influence, ou Détonation sympatique, Essai (Detonation by Influence, or Sympathetic Detonation Test). See Transmission de détonation a distance, known in US as Gap Test

Effet utile. See Potentiel

Épreuve au bloc de plomb par compression (Lead Block Test by Compression). See under Épreuve par compression

Épreuve au bloc de plomb, par expansion (Lead Block Expansion Test) or Épreuve de Trauzl (Trauzl Test). Its French modification is known as Coefficient d'utilisation pratique (CUP or cup) or as Travail spécifique rélatif. See Ref 8, pp 171–74 and Ref 24, p E107-R

Épreuve à la bombe (Bomb Test) or Essai de poudre en vas clos (Test of Propellant in Closed Vessel). See Ref 24, p E108-L

Épreuve (ou Essai) au choc des balles (Bullet Impact Test). See Ref 20, p IX; Ref 8, p 215; Ref 15, p 185; Ref 16, p 195; Ref 18, p 283 and Ref 24, p E108-L

Épreuve au choc de mouton (Shock Test by a Ram). The test with a light weight (such as 2kg) is known as "essai au petit mouton", while test with a heavy weight (such as 30kg) is known as "essai au gros mouton". These tests are listed under Impact (or Shock) Sensitivity Tests in Ref 20, p XVII. See also Ref 24, p E110-L & Ref 9, p 560

Épreuve de combustion (Combustion Test) or Épreuve de l'aptitude à l'inflammation (Capability to Inflame). See Ref 20, p X & Ref 19, pp 329–30

Épreuve par compression [Compression (or Crusher) Tests], used for testing brisance, include: Épreuve des petits plombs (Test by Small Leads), Ref 22, p C492-R; Essai par l'appareil de Chalon (Test by Apparatus of Chalon), Ref 22, p C493-L; and Essai par appareil de Quinan (Test by apparatus of Quinan), Ref 22, p C493-R

Épreuve à la dynamite de pilotis (Test of a Dynamite by Piles). See Ref 24, p E108-R

Épreuve de la finesse des cotons-poudres (Fineness Test for Nitrocelluloses). See Ref 24, p E108-R

Épreuve des petits plombs (Small Lead Blocks Test). See Ref 24, p E109-L & R

Épreuve de la résistance à la chaleur (Resistance to Heat Test). See Ref 20, p XXI & Ref 24, p E110-L. Also described in the Book of Instruction, issued about 1950 by the Commission des Substances Explosives (CSE), Chapitre III, Articles 95–107 and Chapitre IV, Articles 230–31. The book can be obtd from the 2eme Bureau of État Major de l'Armée, Paris, France

Épreuve de la résistance à l'exsudation (Resistance to Exudation Test). See Ref 24, p E110-L

Épreuve de sécurité [Security (or Safety) Test], See Ref 24, p E110-L

Épreuve de la sensibilité à l'amorce (Sensitivity to Primer Test). See Ref 24, p E110-R

Épreuve de la sensibilité à l'inflammation (Sensitivity to Ignition Test). See Ref 24, p E110-R

Épreuves de stabilité des explosifs et des poudres (Stability Tests for Explosives and Propellants). See Ref 24, p E110-R

Épreuve de Taliani (Taliani Test). See Ref 6a, p 837; Ref 11, p 88 and Ref 20, pp XXIV & XXV

Épreuve de tir au mortier ou Essai au mortier éprouvette (Firing Test from Mortar). See Ref 8, p 189; Ref 10, pp 66–7 & Ref 20, p XIX

Épreuve de Trauzl (Trauzl Test). See under Épreuve au bloc de plomb par expansion

Érosion (ou usure) des bouches à feu (Erosion of Gun Barrels). See Ref 24, pp E112-R to E120-R; Ref 8, pp 274–78; Ref 10, p 102 & Ref 13, pp 115–16

Essai avec l'appareil de Quinan (Test With Apparatus of Quinan or Quinan Test). See Ref 8, p 192; Ref 10, p 63 & Ref 22, pp C493-R & C494-L

Essai dans le béton (Concrete Test). It is modification of Essai de Trauzl. See Ref 3, p 105; Ref 4, p 473 & Ref 22, p C495-R

Essai au choc des balles. See Épreuve au choc des balles

Essai de Dautriche pour mesurer la vitesse de détonation (Dautriche's Method for Measurement of Detonation Velocity). See Dautriche, Méthode de

Essai de déflagration [Deflagration (or Ignition) Test]. See Ref 8, p 211; Ref 10, pp 110–11; Ref 11, pp 66 & 83 and Ref 24, pp E135-R & E136-L

ESSAIS d'EXPLOSIFS. Fourteen French official tests devised at the Laboratoire de la Commission des Substances Explosives (CSE) and described in Ref 19, are also described in Ref 24, pp E137-L to E140-R

Essai à la friction (Friction Sensitivity Test). See Ref 8, p 212; Ref 10, pp 75–6; Ref 20, pp XIII–XIV and Ref 24, p E136-L

Essai au mortier éprouvette (Ballistic Mortar Test). See Ref 20, p VII; Ref 8, p 189 & Ref 24, p E136-L & R

Essai au pendule balistique (Ballistic Pendulum Test). See Ref 20, pp VII & VIII; Ref 8, p 269 and Ref 24, p E137-L with Fig E131

Essai au sable (Sand Test). See Ref 20, pp XXI & XXII

Essai dans la terre (Earth Test), known in US as Cratering Test. See Ref 3, p 101; Ref 10, p 67; Ref 13, pp 80–1 and Ref 22, p C554-R, under Cratering Effect Tests

Exsudation, Essais (Exudation Tests). See Ref 4, p 419; Ref 10, p 61; Ref 11, p 65; Ref 19, p 323 and Ref 20, p XI

Exsudation par étuvage, Essai (Exudation on Storing Test). See Ref 24, p E138-L

Galleries d'essais (Galleries for Testing). See Ref 8, pp 235–42; Ref 20, p XIV & Ref 22, p C371-L & R

Marqueyrol, Épreuve de (Marqueyrol Test). See Ref 6, p 178 & Ref 11, p 90

Mesures calorimétriques (Calorimetric Tests). See Ref 20, p IX & Ref 8, pp 60–7

Potentiel ou Effet utile, Essai (Potential or Useful Effect Test). See Ref 10, pp 43 & 64; Ref 13, p 76 & Ref 20, p XX (See also under Pouvoir)

Pouvoir ou Force des explosifs, Essais (Power or Strength of Explosives, Tests). See Ref 20, p XX and as Étude de l'effet utile or Rendement pratique en travail d'un explosif in Ref 8, pp 166–89 & 192–93. The test is also known as Coefficient d'utilisation pratique, Essai

Pressions développées on combustion des poudres, Essai (Pression Developed on Combustion of Propellants). See Ref 8, pp 50–1; Ref 13, pp 73–4 & Ref 20, p XX

Produit caracteristique de Berthelot (Characteristic Product of Berthelot). See Ref 21, p B105, Ref 8, p 53 & Ref 13, p 70

Reprise d'humidité, Essais de (Hygroscopicity Tests). See Ref 19, p 325; Ref 20, p XVI and Ref 24, pp E137-R & E138-L

Sensibilité à l'amorce, Essai (Sensitivity to Priming Test) or Initiating Efficiency of Primers or Detonators. See Ref 10, pp 68–9; Ref 20, p XVIII and Ref 24, p E140-L

Sensibilité au choc du gros mouton (de 30kg) (Sensitivity to Shock With a Big Ram of 30kg which means Impact Sensitivity With 30kg Weight). See Ref 24, p E139-L

Sensibilité au choc du petit mouton (Sensitivity to Shock with Small Ram, which means Impact Sensitivity With Small Weight). See Ref 24, p E139-L

Sensibilité au choc de mouton, Essais (Sensitivity to Shock of a Ram, Tests). Same as Amer Impact (or Shock) Sensitivity Tests. See Ref 8, pp 213–15; Ref 9, pp 560–61; Ref 13, pp 81–3 and Ref 20, p XVII. See also Sensibilité au choc du gros mouton and au choc du petit mouton

Transmission de détonation à distance (Transmission of Detonation at a Distance), known as Aptitude à transmettre la détonation à distance (Ability to Transmit the Detonation at a Distance). See Ref 24, E140-L. Same as Detonation by Influence, Sympathetic Detonation or Gap Test

Transmission de la pression dans un explosif pulverulent (Transmission of Pressure in a Powdered Explosive). See Ref 24, p E139-R

Sensibilité à l'amorce (Sensitivity to Primer). See Ref 24, p E140-L

Travail spécifique relatif (Specific Relative Work) or Coefficient d'utilisation pratique (CUP or cup) (Coefficient of Practical Utilization); corresponds to Trauzl Block Test modified in 1912 by the French Commission des Substances Explosives (CSE). See Ref 24, p E140-L

Vieile, Épreuve de (Vieille Test). See Ref 4, p 661 & Ref 11, p 78

Vitesse de détonation mesurée par la méthode de Dautriche (Detonation Velocity Measured by the Dautriche's Method). See Dautriche, Méthode de (pour mesurer la vitesse de détonation), in this section

Vivacité de combustion des poudres (Quickness of Burning of Propellants). See Ref 13,

pp 90–2 and Ref 24, p E108, under Épreuve à la bombe or Essai de poudre en vase clos (Test in Bomb or Test of Propellant in Closed Vessel)

Refs for French Tests for Explosives and Propellants: 1) É. Sarau, "Théories des Explosifs", Gauthiers-Villars, Paris (1895) 2) F.P. Chalon, Explosifs Modèmes", Ch. Béranger, Paris (1911), 451–54 (Brisance par l' appareil de Chalon) 3) L. Vennin & G. Chesneau, "Les Poudres et Explosifs", Ch. Béranger, Paris (1914), 101–05 (Essais dans le béton et dans la terre) 4) A. Marshall, "Explosives", Vol **2**(1917), 472–73, 478 & 661 5) J. Desmaroux, MP **22**, 245 (1926) 6) M. Marqueyrol, MP **23**, 178 (1928) 6a) J. Goujon, MAF **8**, 837 & 902 (1929) (Épreuve Taliani) 7) E. Burlot, MAF **9**, 799 (1930) 8) L. Vennin, E. Burlot & H. Lécorché, "Les Poudres et Explosifs", Ch. Béranger, Paris (1932), 165 to 279 9) H. Muraour, MAF **12**, 560–61 (1933) 10) J. Pepin Lehalleur, "Traité des Poudres, Explosifs et Artifices", Baillière et Fils, Paris (1935), 59–111 11) J. Reilly, "Explosives, Matches and Fireworks", Van Nostrand, NY (1938), 78–90 12) E. Calvet, Ann Faculté de Sci Marseille **16**, 3–13 (1942) (Detn of brisance by a special apparatus) 14) H. Muraour, "Poudres et Explosifs", Presse Universitaires de France, Paris (1947), 71, 76, 80 & 90 14a) H. Muraour, et al, MAF **22**, 517 (1948) (Essai en vase clos) 15) E. Burlot, MAF **23**, 185 (1949) (Essai au choc des balles) 16) L. Médard & (?) Cessat, MAF **23**, 195 (1949) (Essai au choc des balles) 17) M. Dutour, MP **31**, 74 (1949) (Coefficient de self-excitation) 17a) P. Tavernier, MP **31**, 198–99 (1949) (Gravimetric and loading densities) 18) A. Le Roux, MP **33**, 283 (1951) (Essai au choc des balles) 19) L. Médard, MP **33**, 323–55 (1951) (Fourteen official tests). See Ref 24, pp E135 to E140 20) "Encyclopedia of Explosives and Related Items", PATR **2700**, Vol **1**(1960), pp VII to XXVI 21) Ibid, Vol **2**(1962), p B299-R (Brisance Test) 22) Ibid, Vol **3**(1966), p C311-R (Dautriche Method); pp C492 to C494 (Compression Tests) 23) Ibid, Vol **4**(1969). No French tests are described 24) Ibid, Vol **5**(1972), pp E107-R to E110-R (Épreuves); pp E135-R to E140-R (Essais, French Official)

French Vergé Gelatin. See Vol **5** of Encycl, p D1291, Table 1

French Weapons of WWII. We are listing them (See Table F19 taken from Ref 1) because it is presumed that some of them are still used. No nation (except the US) can afford to change models every few years. Calibers of Fr weapons are in millimeters and abbr Mle is for Modèle (Model)

Table F19

French Weapons of WWII

German Name	*Original Nomenclature*
2.5-cm Pak 34 (f)	25 Mle 1934
2.5-cm Pak 113 (f)	25 Mle 1937
3.7-cm Kw.K.143 (f)	37 SA de char Mle 1938
3.7-cm Kw.K.144 (f)	37 SA de char Mle 1918
4.7-cm Kw.K.173 (f)	47 de char Mle 1935
4.7-cm Pak 181 (f)	47 Mle 1937
4.7-cm Pak 183 (f)	47 Mle 1939
7.5-cm K.231 (f)	75 Mle 1897
7.5-cm K.232 (f)	75 Mle 1897/33
7.5-cm Geb.K.238 (f)	75 M Mle 1928
7.5-cm Kw.K.251 (f)	frz S.A.
10.5-cm K.331 (f)	105 L Mle 1913
10.5 cm K.332 (f)	105 L Mle 1936S
10.5-cm l.F.H.324 (f)	105 C 1934 S
10.5-cm l.F.H.325 (f)	105 C 1935 B
14.5-cm K.405 (f)	145 Mle 1916
15.5-cm s.F.H.414 (f)	155 C Mle 1917
15.5-cm s.F.H.415 (f)	155 C Mle 1915C
15.5-cm K.416 (f)	155 L Mle 1917
15.5-cm K.417 (f)	frz G.P.F. CA
15.5-cm K.418 (f)	155 G.P.F.
15.5-cm K.419 (f)	155 G.P.F.-T
15.5-cm K.420 (f)	155 Mle 1916
15.5-cm K.422 (f)	155 L Mle 1877-1914
15.5-cm K.425 (f)	155 L Mle 1918
15.5-cm K. G.P.F. (f) Ortsfest	
16.4-cm K.(E) 453 (f)	164 Mle
16.4-cm K.(E) 454 (f)	164 Mle 93/96
19.4-cm K.70/93 (E) (f)	194 Mle 70/93
19.4-cm K.G.P.F. (f)	194 G.P.F.
19.4-cm K.485 (f) (Sfl)	194 G.P.F. sur chenilles
22-cm Morser 531 (f)	220 C Mle 1916
22-cm K.532 (f)	220 L Mle 1917
24-cm K.(E) 557 (f)	240 Mle 93/96 M
27.4-cm K.(E) 592 (f)	274 Mle 17
28-cm Mrs.601 (f)	Mortier 280 14/16
28-cm Mrs.602 (f)	Mortier 280 sur chenilles
32-cm K.(E) 657 (f)	320 Mle 70/84 or 70/93
34-cm K.-Gl-(E) 673 (f)	340 Mle 12 (à Glissement)
34-cm K.-W-(E) 674 (f)	340 Mle 12 (à Berceau)
37-cm H.(E) 711 (f)	370 Mle 1915
37-cm K.(E) 714 (f)	370 Mle 75/79
40-cm H.(E) 752 (f)	400 Mle 1915/16
52-cm H.(E) 871 (f)	520 Mle 18

The following French WWII mines and traps (mines et mine-pièges) are listed in Ref 3, pp 206–09:

Table F20

Designation	Overall Height	Overall Length	Overall Width	Type of Filling	Wt of Filling	Total Wt	Igniter
60mm Model 1939 Antipersonnel Mine	8.25"	Unknown	Unknown	Mélinite	5 oz	5.5 lb	Unknown
Light Antitank Mine	4.4	12-11"	8.11"	Same	Unknown	13.4	Pressure type
Heavy Antitank Mine	4.75	16.2	9.85	Same	3.25 lb	27	Pressure type

For initiating the mines and traps, the following devices are listed in Ref 3 pp 211–213, under the title "French Igniters":
1) Rupture Igniter (p 211)
2) Pull Igniter Model 1939 (p 211)
3) Push and Pull Igniters (p 212–13)

We have no information about weapons used after WWII by the French, but presume that they are still using some of the WWII items
Refs: 1) Anon, FAJ (Field Artillery Journal) **34**, 641 (1944) 2) G.B. Jarrett, FAJ **35**, 669 (1945) 3) Anon, "Italian and French Explosive Ordnance", OP (Ordnance Pamphlet) **1668** (1946), pp 206–09

Frequency Tailored Transducers. These ICP (Integrated-Circuit-Piezoelectric) transducers, intended for shock wave, blast, and other expl measurements, are claimed to produce microsecond rise time with high damping to produce clean readable results in instantaneous pressure measurements. Available in ranges to 100000psi, they feature acceleration compensation, 500000Hz frequency response and a built-in IC amplifier to provide a high voltage low output impedance signal. Low Impedance outputs can be operated thru standard coaxial cable without noise or attenuation problems

Frequency tailoring is said to be accomplished by extensive shock wave testing during production and final test to assure good dynamic response from each transducer produced. It also avoids rectification errors (no-symmetrical clipping) in signal conditioning electronics. The output signal approaches that of a non-resonant transducer while retaining the versatility and capability of measuring long duration events
Refs: 1) Technical Bulletin No **11370**, PCB Piezatronics, Inc, PO Box 33, Buffalo, NY 14225 2) Expls&Pyrots **3**(5)(1970)

Fresenius, Karl Remigus (1818–1897). A German chemist noted for his work on analytical chemistry. Designed a desiccator with bell-shaped cover and a nitrogen bulb in the shape of a conical flask with side-tubes having two bulbs near the base
Ref: Hackh's Dict (1944), p 357-R

Fresnel, Augustin Jean (1788–1827). A French physicist noted for his work on optics, such as aberration of light, interference, wave theory of light, etc. He constructed the first practical "interferometer", which is used at present in modified form in Ordnance, such as "interferometric analysis of air flow about projectiles in free flight"
Refs: 1) Encyclopedia Britannica, Vol **9** (1929), p 836 2) Hackh's Dict (1944), p 357-R

Frettage. A French term for the process of constructing a "built-up gun" by means of driving on its barrel heated steel circular bands or rings (hoops). These hoops shrank on cooling and thus hold the walls of barrel tight. Other methods to construct built-up guns are described in Ref 2
Refs: 1) A.M. Taube, "French-Russian Military Dictionary", Voyenisdat, Moscow (1960), p 347-R 2) Encyclopedia of Explosives, PATR **2700**, Vol **2**(1962), p B321-R

Friction and Lubrication (Frottement et Lubrication, in French). *Friction* is the act of rubbing the surface of one body against that of another. It may also be called resistance to relative motion between two bodies in contact. In the case of two solids in contact, with the surface of one sliding over that of the other, the name friction is applied to the force which resists the relative motion of the two bodies. A number (f) indicating the relation of the force (F) just to move an object along a horizontal plane under a pressure (N) is called *friction coefficient:* $f = F/N$

Sliding friction is caused by inequalities in the surfaces and depends on the materials of the substance, but not on the velocity or area of the surfaces in contact

In the case of liquids each particle rubs against the other and this is called *internal fluid friction* or *viscosity*

In order to eliminate or diminish the friction, so-called *lubricants* are used. These substances (auch as oils, fats, greases or finely pulverized materials like talcum) possess the property to make the surfaces slippery when interposed between moving parts. *Lubrication* is the act of making the rubbing surfaces slippery by applying a lubricant
Refs: 1) M.D. Hursey, "Theory of Lubrication", Wiley, NY (1938) 2) R. Sutterlin, MAF **18**, 325–44 (Étude générale sur le frottement) 3) E.D. Tingle, "Fundamental Work on Friction, Lubrication and Wear in Germany", BIOS (BritIntelligenceObjectivesSubcommittee) Rept **1610**(1947) 4) F.P. Bowden & D. Tabor, "Friction and Lubrication of Solids", Oxford Univ Press, London (1950) 5) S.A. Starling & A.J. Woodall, "Physics)), Longmans, Green & Co, London (1950), pp 85–6 6) R.E. Kirk & D.F. Othmer, Edits, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **8**(1952), pp 495–540 (Lubrication and Lubricants) (92 refs) and 2nd Edition, Vol **10** (1966), pp 124–34 (Friction); **12**(1967), pp 557–616 (Lubrication) 7) H.H. Zuidema, "Performance of Lubricating Oils" (ACS Monograph **113**, Reinhold, NY (1952)

Frictional Impact Test. See under FRICTION SENSITIVITY TESTS in Vol **1**, p XIII

Friction Composition for Blasting Fuse. See Vol **4**, p D1071-L

Friction Compositions for Friction Detonators; Friction Firing Devices and Friction Igniters or Primers. These are powders which are easily ignited by friction. Some of the simplest mixtures are K chlorate with sugar or with antimony sulfide. Davis (Ref 1, p 453) gives table listing three compns which have been widely used in friction primers for artillery, separate-loaded ammunition

Table F21

Component	No 1	No 2	No 3
Potassium chlorate	66.67	56.2	44.6
Antimony sulfide	33.33	24.6	44.6
Sulfur	–	9.0	3.6
Meal powder	–	–	3.6
Ground glass	–	10.2	3.6

All the materials are in a powdered condition except the No 1 mixture, where half of the K chlorate is powdered and half of it is granular. The sulfur, which is contd in No 2 and No 3 mixts, makes them more sensitive, but also may make them turn sour after wet-mixing of the components. In order to prevent this, small amts of antacids (such as Ca

carbonate or trimethylamine) should be incorporated. All three mixts are wet-mixed with 5% gum-arabic soln, loaded wet, and dried out *in situ* to form pellets (Ref 1, pp 453–54). Two of these mixts (No 2 & No 3) are listed in Ref 2, p D758-R as "match mixtures"

Many of the compositions suitable for use in Percussion Primers (such as listed in Ref 3, pp D792-R to D805-R) may be ignited not only by percussion but also by friction. See also Instantaneous Percussion Fuze Primers, pp D852-L to D854-L

Refs: 1) Davis (1943), 453–54 2) Anon, "Military Pyrotechnics. Theory and Application", AMCP **706–185**(April 1967), 5–45 3) Encycl of Expls, PATR **2700**, Vol **4**(1969), pp D758-R, D792-R, D805-R and D852-L to D854-L

Friction Detonators. The following types of devices intended for initiating demolition charges, land mines and some underwater charges are described in Vol **4** of Encycl: Friction Detonator, 8-Second Delay, M2 (p D804-R & Fig 67) and Friction Detonator, 15-Second Delay, M1 (p D805-R & Fig 68)

French Friction Detonators are in Ref

Ref: Société Alsasienne d'Explosifs, FrP 102355 & CA **52**, 3346(1958)

Friction Element, Moisture Resistant. A moisture resistant friction element is prepd from a coating compn comprising 100 parts of amorphous P, Sb, and ground glass in 75 parts of plasticized polystyrene emulsion

Ref: E. Daley, USP 2635954(1953) & CA **48**, 5502(1954)

Friction Firing Devices. One Pull-Friction Type Firing Device, M2, is described in Vol **4**, pp D770-R & D771-L & R with Fig 26. It has been used for firing booby traps

Friction; Heat and Percussion in Relation to. Expts on friction and percussion sensitivity, and on sensitization by heat vs sensitization by mechanical shock indicate that a greater volume of Hg Fulminate than of Pb Azide is reqd for build-up of deton. The hot spots due to grit generate the deton wave more easily in Azide than in Fulminate. Percussion sensitivity is apparently more complex involving, in addn to the formation of hot spots thru friction, a fribo-chemical reaction in which there is a more direct transfer of mechanical to activation energy than is the case when the mechanical energy is converted into heat

Ref: J.L. Copp, et al, TransRoySoc(London) **A241**, 197–296(1948) & CA **42**, 7984(1948)

Friction Igniters or Primers (Also known as Friction Tubes). They are devices for the production of fire by the friction of the thrust, either push or pull, of a roughened rod or wire thru a pellet of a special primer composition, such as listed here under Friction Compositions. They are used for firing propelling charges in separate-loaded artillery ammunition. One of such friction primers, M1914, is briefly described in Ref 2, p D795-L

They are sometimes crimped to an end of Bickford fuse for the purpose of lighting it (Ref 1, p 453). Such a device known as Time Blasting Fuse Igniter M1 is described in Ref 2, p D768-R with Fig 19 and on p D762-L

They also are sometimes used for lighting flares, which are thrown overboard from airplanes. For this use, the pull element of the primer is attached to the airplane by a length of twine or wire which is pulled and broken off by the weight of falling flare (See Fig 2-25 in Ref 2, p D957). In one type of the primers for airplane flares, ignition is achieved by pulling a loop of braided wire coated with red phosphorus and shellac thru a pellet made from K chlorate (14 parts) and charcoal (1.6 ps), hardened with dextrin (0.3 p)(Ref 1, pp 453–54). This type of igniter is also listed in Ref 2, pp D758-R & D759-L

Compositions used in friction primers are listed under Friction Compositions (Compare with Friction Detonators and Friction Firing Devices)

Refs: 1) Davis (1943), 453–54 2) Encycl of Expls, PATR **2700**, Vol 4(1969), pp D758-R, D759-R, D762-L, D768-R, D795-L and D957, Fig 2-25 (Aircraft Parachute Flare)

Friction Sensitivity. The importance of standardizing the method and instrument for determining and comparing the sensitivity to friction of expl materials is discussed by Bowden et al (Ref 1) and Kinoshita and Arimura (Ref 2). Bowden points out that, using NG as an example, noexpln occurs until a certain minimal temp is attained from the conversion of mechanical to thermal energy

Refs: 1) F.P. Bowden et al, ProcRoySoc (London) **A188**, 329–49(1947) & CA **41**, 3628d (1947) 2) S. Kinoshita & T. Arimura, Kogyo-KayakuKyokaishi **24**(6), 363–4(1963) & CA **66**, 87219n (1967)

Friction Sensitivity Tests (Qualitative). Two French tests are briefly described in Vol 1 of Encycl, p XIII. They are fully described in Refs 7 & 10 listed on p XIV. One Ger test is briefly described on p XIII and more fully in Refs 1, 2 & 6 listed on p XIV. The Brit test of Dupré is listed on p XIII, but its description is given in BurMinesBull **346**(1931). As this Bull is now out of print, we are giving here a brief description of Dupré test, as given on p 79 of Bull 346

A small amt of the expl was spread on a large stone table and then struck a glancing blow with a mallet made either of rawhide or beech wood. The expl was thus subjected to the combined effect of shock and friction. If the sample exploded, the test was repeated by hitting the expl a glancing blow with a broomstick held at an angle of 60° against the table, taking care that the movement of the stick is in the direction of its axis. If an expln were obtd, the test was repeated on a hard wooden table and finally on a soft wooden one. If an expl, other than those used for caps, detonators, etc exploded, even partly on soft wood, it was considered to be too sensitive for use (Compare with Torpedo Friction Test, described as quantitative test i, Fig F22)

Friction Sensitivity Tests (Quantitative). The following tests are briefly described in Vol 1 of Encycl, pp XIII & XIV:

a) *Rathsburg Test*, using two steel discs, one stationary, another rotating over it. Similar apparatus is described in Ref 1, p 25, Fig 9

b) *US Bureau of Mines Pendulum Friction Apparatus*

c) *Taylor & Rinkenbach's Small Model of Bureau of Mines Pendulum Apparatus*

d) *Impact Friction Pendulum of Spencer Chemical Co*

To these may be added:

e) *Friction Pendulum Test* using apparatus shown in Fig 8 on p 24 of Ref 1, reproduced here as Fig F19. A small, weighed, charge

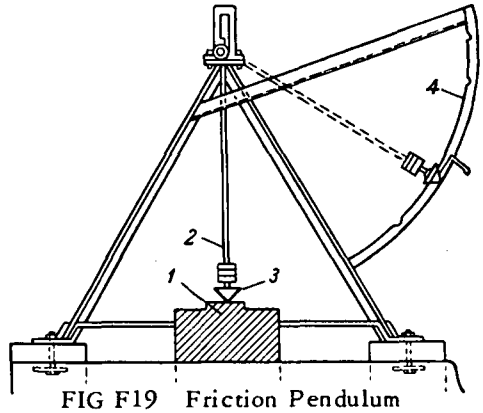


FIG F19 Friction Pendulum

of expl is placed in a depression made in an anvil 1, which can be of steel, bronze, hardwood or of other materials. A steel shoe 2, varying in wt from 1 to 20kg, can be attached to the end of pendulum 3. Then 3 is raised to a predetermined height (indicated on scale 4) and released to swing. If no expln is produced during swinging, a new sample is placed on the anvil and a heavier shoe is used. The test is repeated with heavier shoes until explns are produced. The same results sometimes can be produced by just raising the height of fall of pendulum

f) *Friction Test Using Apparatus of Bowden*, shown in Fig 7 of Ref 2, reproduced here as Fig F20. Here, a thin layer of expl 3, compressed with predetermined pressure, by

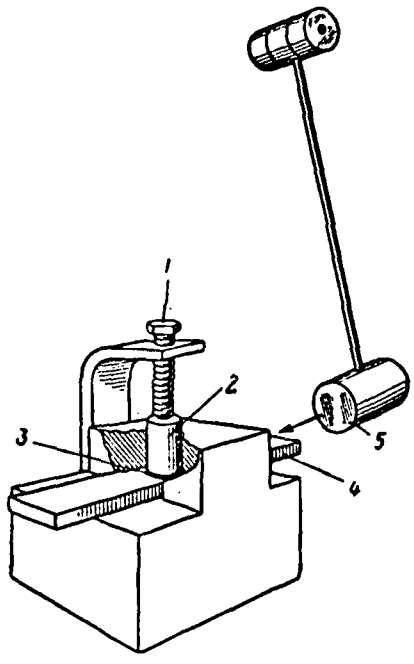


FIG F20 Friction Apparatus of Bowden

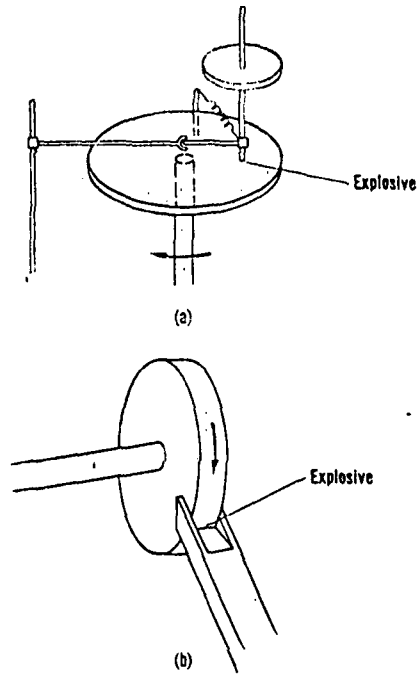


FIG F21 Friction Tests: (a) Liquid explosives (b) Solid explosives

means of screw 1, between a steel roll 2 and sliding plate 4 is subjected to friction when pen dulum 5 hits the plate from a predetermined height. If no expln takes place, the test is repeated with pendulum falling from greater height, etc

g) *Friction Test for Liquid Explosives*, using app shown in Fig 6.6.a on p 73 of Ref 3. Here the expl is smeared on the surface of a rotating disc on which rests a rod of similar or disimilar material, carrying a known wt. The speed of rotation can be varied and also the load employed. The higher the speed and the greater the load before initiation of expl, the lower is the sensitivity of expl. Sometimes an oscillating plate is used instead of a rotating disc

h) *Friction Test for Solid Explosives*, using an app shown in Fig 6.6.b on p 73 of Ref 3, reproduced here as Fig F21. Here, a rotating wheel with rough surface rubs against the sample. The speed of wheel or its pressure against expl can be varied

i) *Torpedo Friction Test*, using an app shown in Fig 6.8 on p 75 of Ref 3, reproduced here as Fig F22. It detemines the effect of a blow which is to some extent at a glancing angle.

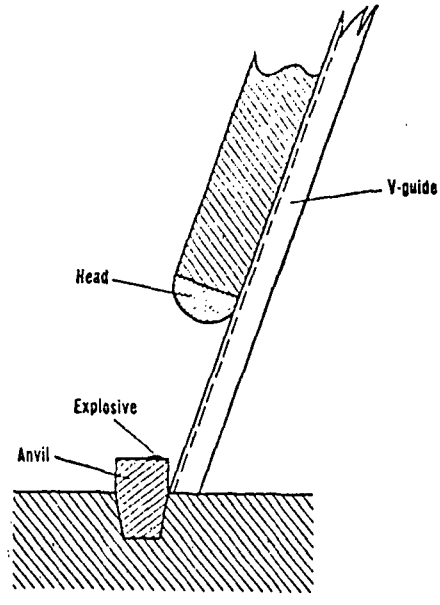


FIG F22 Torpedo Friction Test

This is a common hazard in the handling of expls. In this test, a torpedo weighing 0.5 to 5kg, slides down an inclined plane at an angle 70 or 80° to strike the expl resting on an anvil. The head of the torpedo and the material of the anvil can be varied, as well as the height of fall. Some results of tests are shown in Table 6.1 on p 76

j) *Friction Test Using Apparatus of Bowden-Kozlov*, shown in Fig 10 on p 43 of Ref 4, reproduced here as Fig F23. Here, a small charge of expl 4 is compressed between rolls 1 & 3 by means of piston 5 actuated by hydraulic

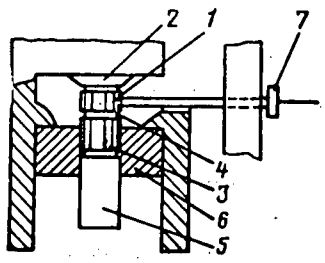


FIG F23 Friction Apparatus of Bowden-Kozlov

press (not shown) and sliding thru cylinder 6. The upper roll 1 slides between support 2 and expl 4 when it is hit by rod with head 7, on being struck by a pendulum (not shown) from a predetermined height. The pressure on piston 5 is varied until 50% of explns are obtd

Refs: 1) Yaremenko & Svetlov (1957), 24–5 2) Gorst (1957), 32–3 (Not in Ref 4) 3) Fordham (1966), 73–5 4) Gorst (1972), 43–4 (Not in Ref 2)

(See also Refs 1 to 15 on p XIV of Vol 1 of Encycl)

Friedel-Crafts Reaction. Any organic reaction brought about by the catalytic action of anhydrous aluminum chloride or related, so-called Lewis acid type catalysts. Discovered in 1877 by C. Friedel and J.M. Crafts, who later uncovered most of the types of reaction such as substitution, isomerization, elimination, cracking, olefin polymerization, addition, etc. Commonly used to displace an aromatic hydrogen atom with an alkyl, aryl or acyl chain

Refs: 1) G.A. Olah, "Friedel-Crafts and Related Reactions", Vol 1–4, Interscience Pub, New York (1963–1965) 2) Kirk & Othmer 10, pp 135–166 (1966)

Friedler's Incendiary Composition consisted of a mixture of crude rubber and metallic sodium or potassium, which was intended to burn while floating on the surface of water

Ref: Daniel (1902), 310

Fringing Groove. During firing, a small amount of copper of the rotating band is forced back behind the band and along the surface of the projectile in the rear of the band. The pressure of the released gas at the muzzle of the weapon and the centrifugal force of rotation combine to throw this excess metal out in a radial direction so that it becomes a fringe around the rear part of the band. When this fringe is excessive and irregular, it builds up air resistance, lessens stability in flight and causes decreased range and decreased accuracy. This excessive fringing can be eliminated if, on machining the band, a groove is cut all around and in the rear of it

Ref: Glossary of Ordn (1959), 124-L

Frittage (Fr). Sintering (Powder Metallurgy)

Front of Detonation Measurements

A photoelectric technique has been devised whereby the position of the deton front in an expl can be measured. The light from the deton front is transmitted by a small diameter optical fiber to the cathode of a photomultiplier tube where the light pulse is converted to an electrical pulse. Additional pulses may be generated by other fibers located elsewhere in the expl. All of the pulses may be recorded on an oscilloscope. The usefulness of the technique has been demonstrated by the measurement of the transmission times of detonators, the propagation velocities of expl columns, the timing of expl systems, the vels

of shock fronts in air, the arrival of shock fronts thru inert materials, and the position of the deton from inside an expl
Refs: 1) W.B. Leslie, "Photoelectric Method for the Measurement of the Arrival of a Detonation Front in an Explosive", Explosivstoffe **18**(7)(1970) 2) Expls&Pyrots **4**(3)(1971)

Front of Detonation, Non-Planar. See Vol **4**, pp D349-R & D350-L

Front of Detonation and Shock Front, also Detonation Zone and Shock Zone. See Vol **4**, pp D350 & D351-L

Front of Reaction in Detonation. See Vol **4**, p D503

Frozen Blasting Gelatin. Blasting Gelatin (See Vol **2**, p B211-R) exists as a yellowish translucent, elastic mass of density about 1.63. It is very difficult to freeze. When frozen, it loses its elasticity and flexibility, and becomes a hard white mass. Unlike Guhr Dynamite and Straight Dynamite, it is more sensitive to shock when frozen than when in a soft and unfrozen state
Refs: 1) Naoúm, NG (1928), 311–12
2) Davis (1943), 343

Frozen Dynamite. All straight Nitroglycerin expls can be frozen. Straight Dynamite when frozen becomes less sensitive to shock and to initiation, but Blasting Gelatin becomes slightly more sensitive. When the expls are afterwards thawed, the NG shows a tendency to exude. Frozen Guhr Dynamite is less sensitive (a drop of more than 1 meter of a kilogram weight or of at least 20cm of a 2kg wt is necessary to explode it). Frozen or unfrozen it can be exploded in a paper cartridge by the impact of a bullet from a military rifle
Refs: 1) Naoúm, NG(1928) 2) Davis (1943) 210, 334, 336

Frozen Gravel Blasting. Underground blasting in permanently frozen gravel was studied in a 7- by 12-foot tunnel by running a nonreplicated 2x2x2 factorial experiment on the effects of type of cut, delay and expl on the fragmentation sub-system. Comparisons were made between a V-cut round and burn-cut round, millisecond delays and half-second delays, using 40% Special Gelatin and 60% High-Density Ammonia Dynamite. None of the differences between delays and expls was notably significant. Results of tests are presented in Refs 1 & 3 and abstracts are given in Refs 2 & 4
Refs: 1) R.A. Dick, "Evaluating Blasting Techniques in Frozen Gravel", MiningCongress-Journal **55**, Sept 1969, pp 30–36 2) Expls-&Pyrots **3**, No 4(1970) 3) R.A. Dick, "Effects of Type of Cut, Delay, and Explosive on Underground Blasting in Frozen Ground", USBurMines Report of Investigation, **RI 7356**, March 1970, 17pp 4) Expls&Pyrots **3**(6), 1970

Frozen Nitroglycerol. NG crystallizes in two forms: a stable form, dipyramidal rhombic crystals which melt or freeze at 13.2–13.5°, and a labile form, glassy-appearing triclinic crystals, mp 1.9–2.2°. It does not freeze readily or quickly. NG in Dynamite freezes in crystals if the expl is stored for a considerable length of time at low temps, its form depending upon the materials with which it is mixed. By virtue of the fact that frozen NG could not be detonated, transportation in the early days of its use was made safer by shipping it in the frozen state
Refs: 1) Naoúm, NG(1928), 110–111 (Freezing and melting of NG) 2) Davis(1943), 207, 212

Fructosans are a group of sugar anhydrides which hydrolyze to *fructose*. One of its derivatives, *Fructosan Trinitrate*, is prepd from fructose
Ref: Hackh's Dict (1944), 358-R

Fructosan Trinitrates. Its alpha form, α-$C_6H_7O_2(ONO_2)_3$; mw 297.14, N 14.14%, OB −24.2%; is produced by the action of mixed acid at 0–15° on d-fructose or on laevulosan; colorless, quickly effluorescing needles from alcohol, which melt at 139–140° and decompose at about 145°. It is readily sol in methyl and ethyl alcohols, acetic acid, and acetone; insol in w. Relatively stable at 50°. It reduces hot Fehling's solution. The alcoholic mother liquors from the α-form yield *fructosan trinitrate* β, crusts of white crystals which melt at 48–52° and decompose at 135°. The material decomposes slowly at 50°. It reduces Fehling's soln rapidly on warming

Refs: 1) Beil **1**, 925 2) Marshall **1**(1917), 197 (Lists the compds as Levulose Trinitrate, α & β and states that they are stable expls) 3) A. Pictet & J. Reilly, HelvChimActa **4**, 613(1921) 4) Davis (1943), 243

Fructose or Laevulose (Levulose, Fruit Sugar, D-(-)-Fructose or Hexose), $C_6H_{12}O_6$; mw 180.16; yellowish white crysts, sp gr 1.669 at 17.5°, mp 103–05°(dec), sol in w, alc & acet. A sugar occurring naturally in fruits and honey; it is the sweetest of the common sugars. Can be prepd by the hydrolysis of inulin or of beet sugar, followed by lime separation. Used in foodstuffs, medicine and as preservative. Can be nitrated by dissolving it in nitric acid, adding sulfuric acid, separating, washing with ice water and purifying by recrystn from alc (See Fructosan Trinitrate, called Levulose Trinitrate by Marshall

Refs: 1) Beil **1**, 918–925 2) Marshall **1** (1917), 197 3) Hackh's Dict (1944), 358-R 4) CondChemDict (1961), 516-R 5) Kirk & Othmer **4**(1964), 137 (Under Carbohydrates)

Fs. Abbreviation for Fuse

FS. Abbr for Fin Stabilized

FS. Chemical agent consisting of a mixture of SO_3 and chlorosulfonic acid used for producing smoke. It has been produced by the US Chemical Corps

Chlorosulfonic acid can be made by reaction of SO_3 and HCl

Ref: A.M. Ball, Ordnance **37**, 430(1952)

Fuchs or Clement Powder. See Clement or Fuchs Powder in Vol **3** of Encycl, p C329-L

Fuel. Any material which produces heat on combustion is known as fuel. Fuels may be divided into *solid* (coal, lignin, coke, wood, sawmill waste, bagasse, bark, straw, and peanut, cottonseed & sunflower seed shells); *liquid* (fuel oils, crude petroleum, kerosene, gasoline, Diesel oil, mazut, coal tar oil, alcohol, benzene and synthetic liquid fuels); *gaseous* (acetylene, blast furnace gas, blue water gas, hydrogen, methane natural gas, oil gas, petroleum gases, producer gas, reformed gas, and commercial butane & propane gases)

Among the solid fuels, only coal & coke are of any importance in the US (See Vol **3** of Encycl, p C352-L for "Coal" and p C391-R for "Coke"); among liquid fuels are petroleum products, especially fuel oils (qv); and among gaseous fuels are natural gases

Besides using coal as a straight fuel, it is used in pulverized state as an ingredient of many commercial expls. They are described in Vol **3** of Encycl, under "Coal Dust and Its Uses", pp C358-L to C359-R. Coal dust was also proposed to be used in "Coal Dust Bomb", briefly described on p C359-R. Coal hydrogenation and coal liquefaction are described under "Coal Processing for Obtaining More Valuable Products", listed on p C379 and in Refs 3, 7 & 11 listed on p C380-R. Among the fuels obtd from coal, may be listed: *coal oil* (p C379-L), *coal tar* (p C379-R). Another solid fuel, *coke*, which is obtd as residue after removing volatile products from bituminous coals (See pp C391-R & C392-L), is used

not only as a straight fuel, but also as a component of commercial expls, as, for example, Dahmenites and Golovine Explosive, listed on p C392-L. Other solid materials used either straight as fuels or as ingredients of explosives are sawdust, charcoal, cereal flours, cellulose, etc

Liquid fuels, such as crude petroleum, mazut, fuel oils, etc are used not only as straight fuels, but also as ingredients of expls, such as AN–FO. They are also used for coating hygroscopic crysts or grains (such as Amm or Na nitrates) used as ingredients of expls. Such coatings also serve as sensitizers for oxidizing salts

Light liquid fuels, such as gasoline, are used in frangible incendiary bombs, in flame throwers, as driving energy of aeroplane and automobile motors, while heavier combustible liquids are used for driving Diesel engines. Methyl- and ethyl alcohols can be added to gasoline in case of its shortage

Beginning before and during WWII many liquid fuels started to be used for propulsion of jet and rocket motors and for spacecraft

Gaseous fuels are used not only as straight fuels, but also for driving engines similar to automobile motors. Due to shortage of gasoline, many automobile engines were converted during WWII in Germany and France to use coal gas.

Sometimes electrical energy when used as a source of heat can be classified as a fuel

Refs: 1) J. Criswold, "Fuels, Combustion and Furnaces", McGraw-Hill, NY (1950) 2) Perry (1950), 150–68 (Solid Fuels); 1568–75 (Liquid Fuels); and 1575–96 (Gaseous Fuels); 4th Edition (1963), 9-2 to 9-6 (Solid Fuels); 9-6 to 9-8 (Liquid Fuels); and 9-8 to 9-11 (Gaseous Fuels) 3) Kirk & Othmer **6**(1951), 892–954 & 960–83 (Fuels); **11**(1953), 2nd Edition **10**(1966), 179–220 (Fuels) 4) Ullmann **4**(1953), 684–721 5) L. Andrussov, Explosivst **1958**, 185 & **1959**, 82–83 6) I.A.M. Poushkin, "The Chemical Composition and Properties of Fuels for Jet Propulsion", Pergamon Press, NY (1962) 7) ASTM Standards for Ash from Petroleum Products, D482–63 (1968) 8) ASTM Standards of Analysis of Coal and Coke Ash, D2795 (1969)

Fuel-Air Explosives. See FAX

Fuel Cells. Accdg to Young & Roselle (Ref 1), the fundamental principles of operation of a fuel cell are essentially the same as that of all galvanic cells. There are two basic half-cell reactions – one involving oxidation and releasing electrons, the other involving reduction and requiring electrons. The two half-cells are separated by an electrolyte barrier so that reaction can occur only by migration of ions. An arrangement of two half-cells is shown in Fig 1 where the fuel

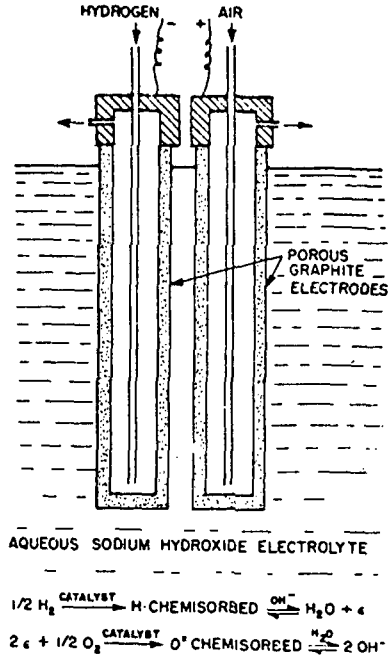


Figure 1 Fuel gas cell operated on hydrogen.

oxidized is hydrogen and the material reduced is the oxygen of the air. The electrolyte is an aqueous soln of Na hydroxide and the hydroxyl ions serve to transport the oxidizing agent to the fuel electrode. Fuel cells, operating at ambient temps and employing fuel gases are capable of a fuel oil efficiency of 70 to

80%, compared with an efficiency of about 35% for a steam plant operating on coal. Other advantages of fuel cells are their high ratio of electrical energy per unit weight, adaptability to both large and small scale applications, and long operating life and storage characteristics. Different types of cells are carbon cells, gas cells and Redox cells. Carbon cells are those in which carbon is used directly as the fuel electrode. For example, in such a cell coal is used as the anode where it is oxidized to carbon dioxide in the half-cell reaction. These cells are inefficient, require high temp (800–1000°C) operation to increase the reaction rate, and at these temps, much of the fuel electrode is converted to carbon dioxide. In a gas cell, fuel gases such as hydrogen or carbon monoxide are used as a fuel. Oxidation of the fuel gases by the electrolyte can be achieved at moderate temps by the use of proper catalysts, and in most cases no side-reactions occur. A *Redox* cell makes indirect use of the fuel as a reducing agent. Electrolyte solns, which are separated by a membrane impermeable to the active ions, contain ions that undergo oxidation and reduction in the cell. The electrolyte solns are regenerated by using carbonaceous fuel for reduction and air for oxidation. The only one considered for commercial development is the fuel gas cell. Table 1 is a summary of several fuel gas sources and applicability in various power sources

Accdg to CondChemDict(1961)(Ref 2) and 1971 edition (Ref 3) the term *fuel cell* can mean rubberized tanks for the storage of liquid fuels and also the following:

Electrochemical devices for the continuous production of electricity by conversion of chemical energy of a continually supplied fuel and oxidant. Like primary cells and storage batteries, fuel cells supply low voltage, direct current energy, but unlike these fuel cells are converters rather than storage devices and produce energy as long as they are supplied with fuel and oxidant. Fuel cells have advantages over storage batteries w/o the necessity for recharging

The principal types of fuel cells under development are:

a) *Hydrox*, utilizing hydrogen fuel and air or oxygen, the reaction product, water, must be continuously removed from the cell, usually by evaporation or condensation. One form uses aq KOH electrolyte, while another employs

Table 1 Fuel Gas Sources

Fuel Gas	Source of fuel gas	Cell applications	Remarks
Hydrogen	Metal hydrides, in particular lithium hydride	Missiles and portable electronic equipment	High fuel cost, favorable weight ratio *ca.* 4#/kw-hr for entire cell including liquid oxygen or peroxide storage
Hydrogen	Commercial tank hydrogen	Lift trucks and tractors, signal equipment, stand-by power sources	Moderate fuel cost, high weight ratio *ca.* 14#/kw-hr for entire cell including compressed oxygen storage
Hydrogen and carbon monoxide	Conventional gasification of coal	Large scale power sources	Impure gases (CO_2 and sulfur compounds), fuel cost using Koppers gasifier and oxygen *ca.* 9 mills/kw-hr
Hydrogen and carbon monoxide	Underground gasification of coal	Large scale power sources	Impure gases, less favorable economically in U.S. than conventional gasification
Hydrogen	Oil refineries (hydrogen produced in cracking and reforming operations)	Moderate sized power sources	Limited application because of restricted nature of fuel source, costs depend on other markets for this source of hydrogen
Hydrogen	Reforming of natural gas	Large scale power sources	Appears favorable in U.S. Southwest, fuel costs *ca.* 4 mills/kw-hr
Hydrogen	Electrolysis of water	Storage of electrical energy	Suggested as a method to relieve peak load problems, not of commercial interest at present
Hydrogen	Radiolysis and photolysis of water	Conversion of other energy forms into electrical energy	No information available

an ion exchange resin as electrolyte. Still another uses natural gas (methane) as the source of hydrogen and molten alkali carbonates as electrolytes

b) *Carbox*, utilizing carbonaceous fuel and oxygen of air. In one example, the electrolyte consists of fused (at 500–800°) alkali carbonates and the electrode reactions involve the interconversion of carbon dioxide and carbonate ions. Carbon dioxide, together with oxygen (or air) is supplied to the cathode and the fuel to the anode

c) *Redox*, in which the electrode reaction involves relatively expensive reactants, which are regenerated externally and reused. In one version, stannous salts are oxidized by bromine in the cell to yield electrical power, while the resulting stannic compds are reduced outside the cell with coal or other carbonaceous fuel. The bromine is regenerated by air oxidation of HBr from the cell

d) *Consumable Electrode*. For special applications a reactive metal such as Na amalgam, may be supplied to a cell as a fuel to provide a controlled source of energy

The most important use of fuel cells is in space vehicles (Apollo series). Liq hydrogen, hydrazine and ammonia have been used as fuel and liq oxygen as the oxidant

Refs: 1) G.J. Young & R.B. Rozelle, "Fuel Cells", JChemEduc **36**(2), 68–73(1959) 2) CondChemDict(1961), 517 3) Ibid (1971), 403-R

Fuel Element. Any material which may undergo the appropriate reaction and be the source of energy in a fusion or fission nuclear reactor is known as *nuclear fuel*. At the present state of technology such fuels are uranium, thorium or plutonium, either as natural materials or enriched or synthesized isotopes of these elements. They are used as solns or as solids (metals, oxides, or carbonates) shaped as plates, rods etc and known as **fuel element** (fuel plate or fuel rod)

Ref: CondChemDict(1961), 315-R; (1971), 403-R

Fuel Oils (FO) and Fuel Containing Explosives. Any liquid or liquefiable product used for generating heat or power, exclusive of oils with flash point below 100°F or oil burning in cotton or wool wick burners (such as kerosene). Oils used in the US may be subdivided into the following grades:

No 1 is liquid with fl p 100°, boiling range 400 to 625°F; used in vaporizing or pot-type burners

No 2 is liquid with fl p above 100°F, distilling at ca 675°F; used for general purposes in domestic heating

No 3 is not known

No 4 is viscous liquid with fl p 130°F; used in industrial burners not equipped with preheaters

No 5 and No 6 are solid which must be liquefied by preheating before burning; used in burners equipped with preheaters. They are sometimes referred to as *bunker fuel oils* and are used in ships, locomotives and industrial power plants

Fuel oils are used extensively in War Plants, as, for example, a typical WWII TNT manufg plant used them in sulfuric acid concentrators, in quadruple effect evaporators of TNT, red waste water, in rotating drums for incineration of the so-called "thick liquor" and as fuel for heating boilers

Some fuel-oils are used for driving internal combustion engines, such as *Diesel Motors*, invented by Ger engineer Rudolf Diesel (1858–1913). Diesel oil is a heavy fuel in which combstn starts by spontaneous ignition due to compression, instead of spark-plugs used in gasoline motors

Diesel oil and other types of fuel oils are used together with oxidizing materials (such as nitrates, chlorates or perchlorates) of which the most important mixtures are **AN–FO** (Ammonium Nitrate–Fuel Oil). These mixts usually contain 5–6% fuel oil and can be prepd on-site or by packing AN first in a borehole, followed by oil. FO's are also used for coating grains of hygroscopic materials, such as Amm or Na nitrates

US Specifications for fuel oils are listed in Refs 5 to 9. AN–FO expls are listed in Addnl Refs

Refs: 1) Perry (1950), 1568–1575 (Liquid Fuels) & 4th Edit (1963), 9–24ff 2) Kirk & Othmer, Vol 6 (1951), 935–54 [Fuels (Internal Combustion)]; 960–83 [Fuels, Synthetic (Liquids)] and 2nd Edit, Vol **14** (1967), 835–67 (Petroleum) 3) R.W. Van Dolah et al; "Further Studies on Ammonium Nitrate–Fuel Oil Compositions", 5thAnnSynp on Mining Research, Nov 19–20 (1959), UnivMissouriSchool-Mining 4) CondChemDict (1961), 517-R & 8th Edit (1971), 403-R 5) US Spec W-F-815C (July 1972) & MIL-F-859E (2) (Aug 1967) (Fuel Oil, Burner) 6) US Spec W-F-800A (May 1968) (Fuel Oil, Diesel) 7) US Spec MIL-F-16884G (March 1973) (Fuel Oil, Diesel, Marine) 8) US Spec MIL-O-1311A (Aug 1955) (Oil, Incendiary, Np, Type 1) 9) US Spec MIL-O-13297 (March 1954) (Oil, Incendiary, 1m, Type 1)

Addnl Refs: A) E.M. Scott, Jr, USP 3016488 (1962) & CA **58**, 407 (1963) (An expl mixt of uncoated AN prills 70 parts, fuel oil 6 and Na nitrate) B) S.R. Brinkley, Jr & G.V. von Elbe, USP 3046887 (1962) & CA **57**, 14046 (1962) (Expl mixt of prilled AN with 2 to 6% FO) C) Z.G. Pozdniakov, Vzryvnoye-Delo, Sbornik No 49/6 (1962) & CA **59**, 2585 (1963) (Russ indust expls based on granulated AN, impregnated with fuel oil and contg some TNT or powd Al) D) K. Hino et al, JapanP 9591 (1963) & CA **60**, 7866 (1964) (AN–FO expls obtd by replacing part of the oil by phenol, cresol, xylenol, aniline, etc) E) R. Kaltenbach, FrP 1368827 (1964) & CA **61**, 15702 (1964) (Expls consisting of porous AN granules with FO absorbed in pores) F) H. Mager, FrP 1370801 (1964) & CA **62**, 3877 (1965) [The disadvantages of on-site mixing of AN & FO can be avoided if the AN (93–96 parts) is kneaded in a mixer with colloidal grease (1–4 parts), such as naphtha or petroleum thickened with stearic acid and glycerol which contains 1–4 ps powd Al, B, Bi or Mg] G) S. Hodgson, CanP 707268 (1965) & CA **63**, 1650 (1965) (Blasting expls prepd by mixing 6 parts of a light FO with 94 ps high density microprills and adding 5 ps of hydratable salt, such as nitrates of Mg, Al, or sulfates of Ca or Mg) H) T. Seguiti, IndMinerari (Rome) **16**, 289–98 (1965) & CA **64**, 7959 (1966) (Uses and properties of AN–oil mixts) I) F. Mezner & S. Uran, Rudasko-MetSbornik **1965** (3–4), p 327 (In Slovenian) & CA **65**, 6987 (1966) (Expl mixt of AN 94.5 & FO 4.5%, called **Nitrol**: pdr of sp gr 1.0 which can be used cartridged or loose) J) J. Dusek & R. Mecir, Rudy (Prague) **14** (3), 72–4 (1966) & CA **65**, 5292 (1966) [The Czechoslovakian expl **DAP–1**, for use in overcast large diam boreholes (>75mm), is prepd by simultaneously adding 95% fuel oil to the borehole directly before blasting without any special mixing] K) K. Stumpf, Glückauf **102** (15), 765–72 (1966) & CA **65**, 11319 (1966) (It is spelled Glueckauf) (Historical review of the expls, based on mixts of AN with org liquids, preferrably Diesel oil) L) G. Hoberstorfer & R.O.R. Oscarsson, SwedP 202595 (1966) & CA **66**, 57493e (1967) (Blasting expl consisting of AN with 5–6% mineral oil are conveyed into a borehole either by compressed air or in a plastic-foil hose) M) J. Kaemmler & J. Ruhmannseder, Rudy (Prague) **14**, 139–42 (1966) (Development of AN–FO expls **Dekammon–1** and **Dekammon–2** at the potash mines of E. Germany). Their compns are not given in CA **67**, 45698h (1967) N) Ch.H. Grant & Th.E. Slykhouse, USP 3377909 (1968) & CA **69**, 20841c (1969) [A 2-component expl of which the 1st mixt consists of AN 94 & fuel oil 6%, while the 2nd mixt consists of AN 37, Al (40–100 mesh) 30, formamide 10, water 12, Karaya gum 1 & Na nitrate 10%. The 1st mixt can be located in the borehole either adjacent to the 2nd mixt or be surrounded by it] O) Fernando S.S. Sobral, Tecnika (Lisbon), **1968** (377), 431–46 (Portug) & CA **70**, 1311j (1969) (A review with 28 refs of properties and methods of application of AN–FO expls) P) B.N. Kukib, VzryvnoyeDelo **1968** (65/22), 269–76 & CA **71**, 31945g (1969) [Comparison of effectiveness in blasting of several Russ expls, showed that **Anfex** (expl contg porous granu-

lated AN & Diesel oil) is superior to **BA-62** (expl contg nonporous AN & Diesel oil)] Q) T. Yamagi et al, JapanP 6918573(1969) & CA **71**, 11402p(1969) [Porous AN granules for use in AN-FO expls, were prepd by spraying high-density particles with 0.3-3.0 wt% aq soln of higher fatty acid salts (such as alkylbenzenesulfonates or alkylaminoacetates), followed by rapid drying at 100°] R) F. Rzepecki, Cement-Wapno-Gips **5**, 148-51 (1969) & CA **71**, 83169b(1969)(New Polish expl **Saletrol** prepd by mixing in-situ in open pit mining of AN 94-96 with mineral oil 6-4%) S) John McKee, BritP 1171536(1969) & CA **72**, 57360h(1970)(AN-FO expls were prepd in a device described in Ref 563 of Vol **5**, pp D1712-L & D1713-L) T) A.I. Tevzadze et al, VzryvnoyeDelo **1969**(67/24), 149-53 & CA **73**, 27144u(1970)(Russ AN/FO expl **Igdanit** contg 2% Diesel oil was more suitable for blasting agate-contg rocks than expl contg 5% oil because it caused less disintegration of rock-surrounding agate. Igdanite 95/5 was, however, more effective because of superior penetrability of oil into AN) U) M. Barbaste, FrAddn 94808(1969) to FrP 1541657 (CA **71**, 51826x); CA **73**, 57647c(1970)(An expl contg AN100, fuel oil 3, naphthalene 2 & Al 4.2g was superior to that contg AN 100, fuel oil 3 & naphthalene 3g) V) J.H. Paasch, SAfricanP 68 05568(1970) & CA **73**, 89729x (1970)(Booster compns for initiation of AN-FO and aq slurried expls contain PETN 50, DNT 10, NC 2, DBuPh 15, oat-hull meal 2 & AN 21%) W) R.E. Schultz & P.H. Rydlund, USP 3540953(1970) & CA **74**, 33218h(1971) (Blasting compn consisting of prilled AN 98-96, Mg nitrate or Ca nitrate in small amts, hydrocarbon oil 1.2-7.2 & carbon black 1.2-7.2 parts) X) L. Nemeth, BanyaszKutIntez-Kozlem **1970**, 14(3), 57-65(Hung) & CA **75**, 8057t(1971)(Improving AN-FO mixts & blasting slurries in Hungary) Y) N.W. Monroe, SAfricanP 6904554(1970) & CA **75**, 65773x (1971)(Expl compns for borehole-blasting agents) Z) J.G.P. Goliger, GerOffen 2021702(1971) & CA **75**, 65776a(1971)(Cord-type expl for underground construction & mining consists of AN granules soaked with extra-light fuel oil. Up to 35% wood powder may be added as a diluent)

Fuel Oils, Analytical. The following determinations were made at US War Plants during WWII: 1) Specific Gravity 2) Moisture 3) Insolubles 4) Flash Point and 5) Pour Point Tests

A detailed description of these tests is given by Clift, Fedoroff & Young in "A Manual for Explosives Laboratories", Vol **3**, Suppl No 2, Chap XIX, pp 1 to 5, Lefax, Inc, Phila (1943)

More recently the tests are described in StdMethodsChemAnalysis 2B(1963), Chapter **40**, "Petroleum and Petroleum Products", pp 1913-2033. Most of these methods are Standards of the American Society for Testing and Materials

Fuel-Oxidizer Type Propellants. See Composite Propellants in Vol **3**, pp C464-L to C474-L

Fuels by Hydrogenation of Coal & Coal Tar. See under "Coal Processing or Obtaining More Valuable Products" in Vol **3** of Encycl, p C379 and also in Refs 3, 7 & 11 listed on p C380-R

Fugacity. Accdg to Hackh's (Ref 1), it is the *escaping tendency* in a heterogeneous mixture, by which a chemical equilibrium responds to altered conditions. In a dilute soln obeying the gas laws, the fugacity equals the osmotic pressure. In other solns it is the value of the pressure for which these equations are still valid

Accdg to Ref 2, fugacity is a quantity which measures the true *escaping tendency* of a gas, a sort of idealized pressure. If primes and double primes refer, respectively, to an ideal gas and a real gas, then:

$dF' = V'dp = RTd\ln p$, and

$dF'' = V''dp = RTd\ln f$,

where dF is a change in *free energy*, or *chemical potential*, produced by a change in pressure, dp; V is the volume of the gas at the absolute temperature T, f is its fugacity and R is the gas constant. Cook (Ref 3) describes a method of calculating fugacities and the calcn of equilibrium concentrations using ratios of fugacities

Refs: 1) Hackh's Dict (1944), 359 2) The Van Nostrand Chemist's Dictionary, NY (1953), 308 3) Cook (1958), 381 (See next item)

Fugacity Determinations of the Products of Detonation were determined by M.A. Cook for PETN, RDX, LNG, Tetryl and 60% Straight Dynamite, by employing the equation of state derived from the hydrodynamic theory and observed velocities of detonation. The so-called reiteration method was developed for solving simultaneously as many equilibria as is necessary to define completely the composition of the products of detonation. Detailed description, together with 14 references is given in the original article:

Refs: 1) M.A. Cook, JChemPhys **15**, 518–24 (1947) 2) Ibid **16**, 1081–86 (1948)

Fugacity of Products. See Vol 4, p D351-L

Fugasnost' or Fugasnoye Deystviye (Rus). Accdg to definition given in Blinov's, "Kurs Artillerii", (Artillery Course), fugasnost' is the destructive action caused by the gases of an explosive and is expressed by the cubic centimeters of expansion products in the cavity of a lead block by exploding a 10g sample. The test is a modification of Trauzl Test, used for detn of power of expls (See Fugasnyi Snariad)

Ref: Fedoroff et al, PATR **2145** (1955), p Rus 7-L

Fugasnyi and Oskolochno-fugasnyi Snariady (Projectiles). *Fugasnyi* [Abbr: Rus letter ф (F)]. It is a high-explosive shell with thin walls and very large bursting charge. It possesses high heaving action and blast effect and for this reason is used against installations as demolition shell

Oskolochno-fugasnyi [Abbr: Rus letters ОФ (OF)]. It is a fragmentation–HE shell with wall thickness and burster chge intermediate between fugasnyi and oskolochnyi shells. It can be used either against personnel or installations.

Oskolochnyi Shell (Abbr O) is a HE fragmentation shell with thick walls and small bursting charge. It is an antipersonnel (A/P) shell

Other Russian projectiles are:

Broneprobivnoy [Б(B)], corresponds to Amer AP (armor-piercing) shell

Broneprozhigayushchii [БЗТ (BP)] means "burning-thru-armor" and corresponds to Amer shaped-charge shells used against the tanks (HEAT Shells)

Granatnyi [Г (G)] corresponds to Amer CP (concrete-piercing) shell

Dymovoy [Д (D)] – Smoke Shell

Zazhigatel'nyi [З(Z)] – Incendiary (I) Shell

Signal'nyi [С(S)] – Signal or Illuminating Shell

Khimicheskii [Х(Kh)]. Chemical (or gas) Shell

Shrapnel' [Ш(Sh)] – Shrapnel Shell

Aghitatsionnyi [А(A)] – Propaganda Shell

Ref: Fedoroff et al, PATR **2145** (1955), p Rus 16 and Figs on pp Rus 14 & 15

Führer of Wien was the first to use pulverized metallic Al as an ingredient of expls. He took British and Belgian patents in 1900

Ref: Daniel (1902), 310

Führer's Silicium Explosive. Mining expl patented by J. Führer in 1904. It was the first expl to contain silicon. Its composition was Amm nitrate 77, DNT 10.5, Si (crystalline) 10 and wood flour. It was not powerful and its oxygen balance was negative

Ref: R. Sartorius, MP **34**, 211 (1952)

Fulgor Powders. Smokeless powders patented in 1895 in England by Ungania, existed in

several varieties, as for example:
Fulgor A (suitable for military purposes): Endecahydronitrocellulose (or hendecahydronitrocellulose) 75.8, Hexanitromannite 22.7 & paraffin 2 parts.. Aniline black 0.05 pts was added. Gelatinized by means of ethyl acetate
Fulgor B (suitable as sporting powder): Endecahydronitrocellulose 100 & $K_4Fe(CN)_6$ 0.05 added. Gelatinized by means of ethyl acetate
Fulgor C (suitable as sporting powder): Heptahydronitrocellulose 64.5, Hexahydronitrocellulose 16.2, $KClO_3$ 12.1, $Ba(NO_3)_2$ 6.4, paraffin 0.8 and $K_4Fe(CN)_6$ 0.05 added. Preparation of endeca- and hepta-nitrocelluloses, as well as of Hexanitromannite, is given in Ref 1
Refs: 1) E. Ungania, BritP 12325 (1895) & JSCI **15**, 376 (1896) 2) Daniel (1902), 311–12 3) Fedoroff & Clift **4** (1946) 37

Fulgurites. Accdg to Daniel (Ref 1) & Gody (Ref 2), they were Hungarian Dynamites consisting of NG 60–90 absorbed by a mixture of wheat flour & Mg carbonate 40–10%. Another Fulgurite was patented in 1900 in France by Serrant. It contained Nitrocompounds of cresol or naphthalene
Refs: 1) Daniel (1902), 313 2) Gody (1907), 362 3) Fedoroff & Clift **4** (1946), 37

Fuller's Earth. A variety of clay-like materials containing silicates of Fe and Mg which have natural adsorptive powers; have been used in industry for many purposes, among them as absorbent for NG in Dynamites
Refs: 1) Davis (1943) 195, 216, 218, 332 & 336 2) Fedoroff & Clift **4** (1946), 37 3) Cond-ChemDict (1961), 517-R

Füllung or Fülpulver (Fp or FP). German name corresponding to expls used as *fillers* for bombs; shells, grenades or mines. They serve as *bursting charges.* A complete list of such expls is given in PATR **2510** (1958), pp Ger 46-R to Ger 48-R, under FILLER

Fulmenit (Ger). Permissible expls, such as a) AN 86.5, NC 4.0, TNT 5.5, charcoal 1.5 & paraffin oil 2.5% (Refs 1, 3 & 4) and b) AN 82.5, NC 4.0, TNT 11.0, charcoal 1.5 & paraffin oil 1.0% (Refs 2 & 4) (See also Wetter-Fulmenit)
Refs: 1) Gody (1907), 595 2) Marshall **1** (1917), 391 3) Barnett (1919), 113 4) Clift & Fedoroff **2** (1943), 0 F3 5) PATR **2510** (1958), p Ger 53-L

Fulmibois. A French name for Nitrolignin or Nitrated wood which was prepd by nitration of sawdust
Ref: Daniel (1902), 313

Fulmicoton. Accdg to Patterson's French-English Dictionary it is **Guncotton**, while *Fulmicoton soluble* is *Pyroxyline.* Guncotton has N content 13.47% and its formula corresponds to Endecanitrocellulose, $C_{24}H_{29}O_9(ONO_2)_{11}$. Its N content is slightly higher than that for CP_1 (Coton-poudre No 1), listed in Vol **3** of Encycl, p C551-R. Pyroxiline contains about 12% N and it is listed as CP_2 (Coton-poudre No 2) on p C551-R

Fulmicoton de Faversham or Tonite. Under these names are known expls consisting of Fulmicoton & Ba nitrate with or w/o other ingredients. The following compns are listed by Daniel (Ref):
Tonite No 1, patented in England in 1874 by Trench, Faure & Mackie contd Fulmicoton 51.6 & Ba nitrate 48.4%; white pdr
Tonite No 2 contd charcoal as the 3rd ingredient; grey pdr
Tonite No 3, patented by Trench contd Fulmicoton 14.55 to 19.0%, m-DNBz 13.2 to 13.0 & Ba nitrate 72.25 to 68.0%. It was manufd in England beginning in 1889 and used extensively for underwater blasting and as a bursting chge for torpedoes. It was manufd in the US by the

Tonite Powder Co of San Francisco

In Belgium, the Tonite of compn Fulmicoton 50, Ba nitrate 40 & K nitrate 10% was manufd at Wetteren

Ref: Daniel (1902), 769–71 (Tonite)

Fulmicoton, soluble (Fr). One of the names for Pyroxyline (NC with N content ca 12.2%)

Fulminant d'argent. Fr for Silver Fulminate, described under Fulminates

Fulminant d'argent (ou L'argent fulminant) de Berthollet. Fr for Fulminating (or Detonating) Silver of Berthollet

Fulminant d'argent de Brugnatelli (ou de Howard). Fr for Silver Fulminate, described under Fulminates

Fulminant d'or. Fr for Fulminating Gold described under Fulminates

Fulminant de platine. Fr for Fulminating Platinum

Fulminate Blasting Cap of Nobel. See under DYNAMITE in Vol 5, p D1588-R

Fulminate Chlorate (90:10). A mixture of MF (Mercuric Fulminate 90 & K chlorate) usually employed in older types of detonators

The following wts of this mixture are required for complete detonations of: TNT 0.25g, Tetryl 0.19, 80:20–TNT:Tetryl 0.21, 50:50–TNT:Tetryl 0.20 (Ref, p 183), TeNA 0.20 and HNDPhA 0.18g (Ref, p 186)

Ref: Davis (1943), 183 & 186

FULMINATES

Fulminates are salts of *fulminic* or *paracyanic acid* (qv), which is isomeric with isocyanic acid. Fulminates should not be confused with *Fulminating Compounds* (qv). Structural formulas of fulminic acid and of its salts have not yet been definitely established. The free fulminic acid has not been isolated from its solns, as it undergoes polymerization very rapidly both in aqueous and in ethereal solns. The acid and its metallic salts, $M(ON{:}C)_n$, are very poisonous and explosive. The best known and most used salt is Mercuric Fulminate (qv)

A historical background of fulminic acid and salts was given in the book of R. Scholl, "Entwicklungsgeschichte der Knallsäure", Habilitat, München & Leipzig (1893) and by M. Giua in "Tratto di Chimica Industriale", 6(1), UTET, Torino (1959), pp 415–19

Fulminic Acid (qv) was prepd and identified in 1800 by E. Howard, who also prepd and identified MF, first prepd (but not identified) in the 17th century by J. Kunkel von Löwenstern. Silver Fulminate was first prepd in 1798 by L. Brugnatelli

More thoroughly, HON:C or simply HONC, and its salts were investigated in this century by Wöhler, Martin et al (Refs 2, 3 & 10)

Alkali Fulminates, MONC of Cs, K, Na & Rb were prepd and investigated before WWII in Warsaw by Hackspill & Schumacher (Ref 18). The Na salt was prepd in 1921 by A. Langhans (Ref 6). It was found that all alkali fulminates have similar properties, such as: they all can be dissolved in methanol crystallizing on its evapn, probably in the hexagonal system. When dry, they were easily detonated by feeble rubbing or by heating. They easily formed expl double salts with MF. For example RbF–MF easily exploded at 45°. Description of K and Na salts are given separately

Ammonium Silver Fulminate, $Ag(NH_4)(ON)_2$; mw 209.96, N 20.02%; crysts (alc+eth); unaffected by light; mp – expl on heating; sl sol in w; sol in alc. Can be prepd by the action

of Amm iodide on SF. It is a less violent expl than SF {Ref 1, p [777]}

Cadmium Fulminate, $Cd(ONC)_2$; mw 196.45, N 14.26%; wh crysts, mp – expl at 215° in 5 sec; sol in w & methanol; sl sol in ethanol. Can be prepd by shaking Cd amalgam with MF dissolved in methanol. Heat of deton 470cal/g. It is a powerful expl resembling MF in its props. It is stable when kept dry but decomp in the presence of moisture. Smallest amts which cause detonation of: Tetryl 0.008g, PA 0.05g, TNT 0.11g, TNAnisol 0.26g & TNXylene 0.35– [Ref 1, p(376) & Ref 23, pp 407 & 411]

Copper (I) Fulminate or Cuprous Fulminate. See Vol 3 of Encycl, p C520-R and Davis (Ref 23, p 407) gives smallest amts of Copper Fulminate which cause detonation of: Tetryl 0.025g, PA 0.08g, TNT 0.15g, TNAnisole 0.32 & TNXylene 0.43g

Copper (I) Fulminate Explosive Complexes, listed below are described in Vol 3 on pp C520-R & C521-L:

Copper(I)-barium Fulminate, $Ba[Cu(ONC)_3]_2 \cdot 4H_2O$

Copper(I)-calcium Fulminate, $Ca[Cu(ONC)_3]_2 \cdot 4H_2O$

Copper(II)-dicopper(I) Fulminate, $Cu[Cu_2(ONC)_4] \cdot 2H_2O$

Copper(I)-disodium Fulminate, $Na_2[Cu(ONO)_3] \cdot 3H_2O$

Copper(I)-sodium Fulminate, $Na[Cu(ONC)_2]$

Copper(I)-strontium Fulminate, $Sr[Cu(ONC)_4] \cdot 2H_2O$

Mercuric Fulminate or Mercury Fulminate [Fulminate de mercure in Fr; Knallquecksilber in Ger; Fulminato di mercurio in Ital; Fulminato de mercurio in Span; Gremuchaya rtut' in Russ; and Raikō or Raisan suigin (Thunder Mercury) in Japan], $Hg(ONC)_2$. Structural formula not established. Pepin Lehalleur (Addnl Ref J, p 141) proposed $Hg(ON{:}C)_2$ and that of Scholl $Hg{<}^{ON:C}_{ON:C}$; mw 284.65, N 9.85%, OB to CO_2 –17% & to CO –5.5%; hydrate $Hg(ON:C)_2 \cdot \frac{1}{2}H_2O$ (crystd from w)(Ref 23, p 410) has mw 293.64, N 9.55%. Anhydrous form(crystd from alc) is white to grey crysts of density 4.42–4.43, exploding at 210° in 5 sec; its apparent density is 1.55 to 1.75; when subjected to pressures of 3, 10 20 & 50 thousand psi, densities of 3.0, 3.6, 4.0 & 4.3 are obtd. Pressures of 25 to 30 thousand psi (or more) cause the desensitization of MF to the extent of becoming *dead-pressed.* Such material merely ignites and burns when subjected to contact with flame, but it can be detonated if it is covered with a layer of loose or sl compressed MF and ignited

MF is sol in 1 liter of w to the extent of: 0.71g at 12°, 1.74 at 49° and 7.7 at 100° (Ref 23, p 410); only sl sol in cold ethanol. It decomp in the presence of aq solns, chlorides, carbonates, hydroxides etc. Due to the presence of small amts of Hg, formed by exposure of MF to light or elevated temps, it readily forms amalgams with Co, brass or bronze, thus components contg these metals must be protectively coated if used with MF. MF is also sol in pyridine and aq NH_3 or K cyanide and can be recovered from pyridine by adding w and from NH_3 & cyanide solns by adding acid

MF reacts with concd HCl to form hydroxylamine, $H_2N.OH$, & formic acid, HCOOH, and it reacts with Na thiosulfate in aq soln to form Hg tetrathionate, accdg to the equation:

$$Hg(ON:C)_2 + 2Na_2S_2O_3 + H_2O \rightarrow HgS_4O_6 + 2NaOH + NaCN + NaNCO$$

This reaction can be used for detn of purity of MF

Preparation of MF

MF was first prepd in the 17th century by Swedish-German alchemist Baron Johann Kunkel von Löwenstern living from 1630 to 1703 (Hackh's Dict gives 1638 as date of birth). He obtd this dangerous expl by treating mercury with nitric acid and alcohol. The method was described in the book of Kunkel published in 1716 after his death. This book is listed and the prepn is briefly described by Davis (Ref 23, p 404) and Pérez Ara (Ref 24, p 541). A brief biography of Kunkel is given in Ref 24, p 541 and in Hackh's (1944), p 474

As no use of Kunkel's expl was found by him and by other alchemists, the compd became forgotten until Edward Howard of England rediscovered it in 1799–1800. He described its prepn in PhilTransRoySoc **1800**, 204, and his method of prepn is given in Davis (Ref 23, p 404). Howard examined props of MF but it does seem that he proposed its use as a *percussion initiator* of BkPdr. This honor belonged to the Scottish Clergyman Alexander Forsyth who patented in 1807 in England such a device. The history of this and of later development of MF initiators and of various types of cartridges is described in Vol 5 of Encycl, p D755-R & D756-L. More important application of MF was in A. Nobel's Blasting Cap invented in 1867. This cap was used for detonation of NG and Dynamite (See "Fulminate Blasting Cap" in Vol 5 of Encycl, p D1588-R)

Besides describing Howard's method, Davis (Ref 23) described on p 406 a laboratory method using 5g of Hg. We found that it is safer to start with 2g Hg and to proceed as follows:

Laboratory Preparation:

1) 2g of pure redistd Hg is added to 10ml of pure nitric acid (sp gr 1.42) in a 100-ml Erlenmeyer placed under a hood and left there without agitation until complete dissolution

2) The resulting soln of Hg nitrate in excess of acid is poured into 20ml of 90% alcohol contd in the 2nd 100-ml flask, which is placed in a trough of running water located under a hood and behind a safety glass or plastic. A vigorous reaction results, accompanied first by evoln of white fumes, then by brownish-red and finally again by white fumes. At the same time crysts of MF deposit on the bottom of the flask. After about 20 mins, the fuming ceases and the contents of flask are poured into a 250-ml beaker contg cold w. After the bulk of acid w has been removed by decantation, the crysts are transferred onto a Büchner connected with suction flask to be washed with w until it becomes neutral to litmus. This gives about 3.4g of crude greyish (due to presence of small amt of metallic Hg) product, which corresponds to good-grade commercial product. It can be stored under water until a sample is required for exptl purposes. Then part of product is dried in a desiccator

MF has been thoroughly investigated especially before and during WWI by Col Solonina of Russia (Addnl Ref A). Other investigators were Liebig, Chandelon, Bechmann, Tschelzof (Chel'tsoff), Wöhler, Martin, Langhans & Stettbacher (Ref 17, p 141)

Pepin Lehalleur (Ref 17, pp 143–44) described Liebig's and Chandelon's methods of manuf which have been used in France

Pérez Ara (Ref 24, p 545) briefly describes an industrial method using 1 kg of Hg, 9kg of nitric acid and 8–12kg of alcohol. He also describes on p 546 industrial methods of Bechmann, de Bruyn and of the Troisdorf Fabrik of DAG in Germany. All of these methods differ only slightly from the US method described below. The method of de Bruyn, instead of adding acidic Hg nitrate to alc, bubbles vapors of alc thru the soln of Hg in nitric acid. The reaction proceeds at 70°. In the method of the Troisdorf Fabrik, small quantities of Zn & Cu are added during dissoln of Hg in nitric acid (2g of Zn & 3g of Cu per 600g Hg and 5kg nitric acid). This method is claimed to give white MF

In all methods, except that of Troisdorf, the resulting MF is of greyish color due to the presence of small quantity of free Hg. Such MF is not suitable for loading into caps made of Cu or Cu-contg alloys such as brass or bronze. This is because free Hg attacks Cu forming a Cu amalgam, and this will react with MF to form Cuprous Fulminate which is less stable and more sensitive than MF

In order to avoid the formation during manuf of greyish MF, Solonina proposed adding 1.5g of $CuCl_2$ per each 50g of Hg during its dissoln in nitric acid, whereas Adler proposed dissolving in nitric acid a small quantity of Zn before adding Hg (4g Zn per 50g Hg)

Accdg to Davis (Ref 23, p 407), MF can also be prepd by replacing ethanol with acetaldehyde, paraldehyde, metaldehyde or dimethyl- and ethylacetal. Another method consists of

treating the Na salt of nitromethane with an aq soln of $HgCl_2$ at 0° to form a wh ppt of mercuric nitromethanate. This gradually becomes yel and when digested with warm dil HCl yields MF

MF can also be manufd by Biazzi Process. Since it is not described in Vol 3 of this Encycl, pp C501ff, under "Continuous Methods for Manufacturing Explosives" it is suggested that info be obtained directly from Dr Mario Biazzi, Ing, Vevey, Switzerland

Manufacture of MF in the USA

The following method used to be employed at Picatinny Arsenal and probably is still used in other US plants because a similar method is described in Ref 32 and Ref 35 (1967)

In order to produce MF of quality complying with requirements of US Military Specification JAN-M-219, all raw materials: mercury, nitric acid and ethanol should be of high purity

Procedure:

1) About 1 lb of pure, redistilled mercury was carefully added to an open earthenware vessel (or a 5 liter flask) contg 8 to 10 lbs nitric acid (d 1.4), which was in excess of the amount required by the equation: $3Hg+8HNO_3 \rightarrow 3Hg(NO_3)_2+2NO+4H_2O$

A large number of such charges were usually prepared and allowed to stand overnight until the Hg was completely dissolved

2) A 10 gallon Pyrex flask or balloon, contg 8–10 lbs of 95% alcohol and fitted with a reflux condenser, was placed on a rack in a trough with running cold water. This installation had to be either outside or in a building provided with an exhaust system

3) With the condenser removed, the acidic soln of mercuric nitrate was poured into the alcohol and then the condenser was replaced. After about 2–3 minutes, a violent reaction started and the liquid boiled with evoln of white fumes. Most of these fumes were recovered by the condenser, however. As the reaction approached the end point, the color of the fumes usually changed to brownish-red due to the decomposition of nitric acid by heat. As this heat might also decompose the MF (which had just formed), a small quantity of dilute alcohol had to be added at this point to moderate the reaction. The reaction normally lasted about 1½ hours and MF crystallized

5) After cooling the charge in the flask, it was dumped onto a cloth screen and the crystals of MF washed with cold running water to remove excess nitric acid and impurities known as "fulminate mud"

6) The washed MF was then drained and packed into cloth bags which were stored in tanks of water until required for use

For shipment, the bags were packed in barrels of wet sawdust

7) When required for use, the MF was removed from the bag and spread on a cloth in a thin layer and dried in a special building where the temp was carefully regulated to 43°(110°F)

8) The acid mother liquor and the water washings were neutralized with alkali, evaporated to recover the alcohol and treated to recover any Hg or its salts

The usual *yield* was about 1.25 parts of slightly greyish MF per one part of Hg. Its purity was 98–99%, which corresponded to usual commercial product (Ref 32, p 588 & Ref 35, pp 7-5 & 7-6)

The intermediate products of oxidation an and nitration involved in the prepn of MF are as follows:

$CH_3.CH_2OH \rightarrow CH_3.CHO \rightarrow CH_2(NO).CHC \rightarrow CH(:NOH).CHO \rightarrow$
Ethanol — Acetaldehyde — Nitrosoacetaldehyde — Isonitrosoacetaldehyde

$CH(:NOH).COOH \rightarrow C(NO_2)(:NOH).COOH \rightarrow O_2H.CH:NOH \rightarrow$
Isonitrosoacetic acid — Nitroisonitrosoacetic acid — Formonitrolic acid

$C:NOH \rightarrow Hg(ONC)_2$
Fulminic acid — Mercury fulminate

(Ref 35, p 7-6 & Ref 23, p 407)

Accdg to Ref 35, p 7-6, the above commercial product can be purified to 99.75% by dissolving it in concd Amm hydroxide, filtering the soln, cooling the filtrate, and reprecipitating slowly, adding concd nitric acid with rapid agitation, while keeping the temp below

35°. The pptd pure MF is washed with distd w until free of acid. The yield is 80–87%

Accdg to Ref 17, p 146, a fairly pure MF can be obtd by rinsing the crude MF (packed on two layers of filter paper placed in a Büchner) with several portions of 80–85% ethanol, thoroughly removing by suction each rinsing and testing it by diluting with w. The completion of rinsing is indicated by absence of turbidity on addn of w

There are two other methods of purification listed in Ref 17, p 146. Steiner dissolved crude MF in concd aq soln of KCN followed by adding concd HCl and washing the resulting ppt with w. Solonina dissolved 1 part of crude MF in 7 parts of pyridine, filtered the soln and poured it into 100 parts of cold w

The size of the crysts of MF is an important factor. While very finely divided MF (consisting mostly of fragments of crysts) is undesirable because the crysts might contain large amt of impurities, thus diminishing the efficiency of MF, too large crysts are also not desirable because they are very sensitive to friction and shock. Therefore, the US Military Specifications such as JAN-M-219, provide a certain minimum and maximum size for crysts. Fig F24 reproduced from Ref 35, p 7-5, shows typical crysts magnified 125 times. The Fig 94 on p 409 of Ref 23 (not reproduced here) shows crysts for use in primer compns, magnified 35 times

FIG F24 MF Crystals

If crysts of MF are so small that they look like colloidal particles, the material is very difficult to initiate even if it is nearly 100% pure

Explosive and Other Properties of MF (Refs 5, 7, 9, 11, 12a, 15, 16, 17, 21, 22, 23, 24, 25, 26, 27, 28, 29, 30, 32, 33, **34**, 35, 37 & 38) (Also Addnl Refs F, R, S, T & X):

Activation Energy. 29.81kcal/mol; Induction Period 0.5–10 sec (Ref 34)

Ballistic Mortar Test – not found

Behavior Towards Flame. When a single cryst is ignited, it burns rapidly with a flash, but does not detonate; when a layer of crysts is ignited, a high order of deton takes place

Brisance by Sand Test in 200g Bomb. 0.4g MF crushes 17.9 to 23.4g sand compared with 48g for TNT, which means 27.3 to 59% of TNT. It is greater than LA and smaller than DADNPh.

Compatibility with Metals. When dry – reacts rapidly with Al & Mg and slowly with Cu, Zn, brass & bronze. When wet – reacts immediately with Al & Mg and rapidly with Cu, Zn, brass & bronze. Dry or wet does not attack iron or steel (Ref 34)

Destruction of MF can be achieved by adding it, with stirring, to at least 10 times its weight of 20% Na thiosulfate. Some poisonous cyanogen gas may be evolved (Ref 34)

Deterioration of MF in Storage at 10–20, 30–35, 50 and 80°. See under Stability of MF in Storage

Detonation Rate for Pressed MF. 3500m/sec at d 2.0, 4250 at 3.0 & 5000 at 4.0 (Ref 34)

Electric Spark; Sensitivity to. MF is capable of being ignited by a spark from a person charged to less than 5000 volts, a condition that is possible in plant operations. Brown et al (Ref 27) give for Sensitivity to Electrostatic Discharge 0.025 joules (Ref 34)

Explosion (or Ignition) Temperature. 210° (in 5 sec) 239° in 1 sec & 263° in 0.1 sec (no cap used) (Ref 34)

Flammability Index – not found

Friction Pendulum Test. Expl on fiber & steel shoes (Ref 34)
Gas Volume Evolved on Explosion – 243cc/g (Ref 34)
Heat of Combustion. 938cal/g
Heat of Explosion. 427cal/g
Heat of Formation. 226cal/g
Heat Test at 100°. Explodes in 16 hrs (Ref 34)
Hygroscopicity. Gains 0.02% at 30° % 90% RH (Ref 34)
Impact Sensitivity Tests. Bur Mines App – 5cm for 2kg wt (20mg sample); Pic Arsn App – 4 inches for 1 lb wt (30mg sample) (Ref 34). It is more sensitive than LA of LStyphnate (Ref 35)
Initiating Efficiency of MF. Although MF is not as efficient an initiator as LA or DAzDNPh, it is satisfactory when used in conjunction with Tetryl, RDX or PETN boosters. During the 1st half century MF was almost the only initial detonating agent used and its efficiency was considered satisfactory (Ref 35)
Initiating Efficiency. Grams of MF Required to Give Complete Initiation: 0.25 for TNT, 0.20 for Tetryl, 0.19 for RDX and 0.17 for PETN (Ref 34). Also Expl D 0.85, TeN-Aniline 0.25 and PA 0.21. Rather different values are given by Davis (Ref 23, p 407): Tetryl 0.29, PA 0.30, TNT 0.36, TNAnisole 0.37 & TNXylene 0.40g
Initiating Sensitivity to Electrostatic Discharge. See under Electric Spark; Sensitivity to
International Heat Test (75°). Loss in wt in 48 hrs 0.18% (Ref 34)
Loading Into Blasting Caps pressed at 3000 psi (Ref 23, p 410)
Plate Dent Test – not found
Power (or Strength) by Trauzl Lead Block Test. 37 to 50% TNT (Addnl Ref T); 51% (Refs 12a & 34). It is more powerful by Trauzl Test than LA (Ref 35)
Rate of Detonation. See Detonation Rate
Rifle Bullet Impact Test – not found
Sand Test. See under Brisance
Sensitivity to Electric Spark or to Electrostatic Discharge. See under Electric Spark
Sensitivity to Initiation – not found
Sensitivity to Pressure. When pressed at pressures in excess of 25000psi, MF will become *dead-pressed,* which means that it will not detonate either by flame, percussion or stab action. When such MF is ignited by heat or flame (as happens in electric detonators), it will burn but not detonate. However, it is possible to detonate the dead-pressed material by means of a strong Blasting Cap or by covering it with loose (or sl pressed MF) and then igniting the ensemble. In these cases the velocity of deton is even higher than for material that has not been dead-pressed (Ref 35 & Addnl-Ref T)
Specific Heat. 1.1cal/g/°C (Ref 34)
Stability. Accdg to Ref 35, p 7-7, the relatively poor stability of MF has been its most disadvantageous characteristic and the main reason for efforts to replace it with a superior substitute. The usual stability tests are only applicable to temps up to about 85° due to expln in a relatively short time at temps above 85°. When MF deteriorates, it is chiefly with the production of a non-explosive solid rather than gaseous products of decompn. It has been found that when its purity has been reduced to approx 92%, the initiating efficiency is practically destroyed, although the material will explode when ignited. When purity has been reduced to 95%, the stability is considerably impaired. (*Note:* Because of the poor stability of MF, it is no longer permitted by the US Military for service use)
Stability of MF in Storage. MF of purity 99.75% has been found to require storage at 50° for 24 months to become deteriorated to 92% (Ref 35, p 7-7). Different results are given in Ref 34, p 203, where MF of 99.75% purity deteriorated in 18 mos to 94.81%, while 98.86% deteriorated at the same period to 79.99%. Still other results are given in Table 7-3 on p 7-7 of Ref 35. Here, after storage at 50° for 8 months, the purity decreased to 95% and after 11 mos to 92%. When stored at 80°, purity dropped to 95% in half a day and to 92% in one day. When stored at 30–35° purity dropped to 95% in 1.7 years and to 92% in 5.8 years. When stored at 10–20°, a purity of 95% was

obtd in 7–8 years, while 92% was obtd after 9–10 years

Stab Sensitivities at Various Densities are given in Table listed on p 202 of Ref 34

Storage. MF is stored under water and transported in a wet condition, because in the dry state it is extremely sensitive to any mechanical action. In winter it is stored in 50/50–water/methanol or water/ethanol. MF should never be stored together with any other expl(s) because the expln of even a relatively small amt of MF present in a dried state might cause the detonation of wet MF and other expls. If any of the material contg MF is spilled on the floor or table, it must be destroyed by washing with a satd soln of Na thiosulfate (Ref 35)

Thermal Conductivity. $1x10^{-4}$cal/sec/cm/°C (Ref 34)

Thermal Stability. See Heat Test at 100°, International Test (75°) and Vacuum Stability Test at 100°

Toxicity of MF. Mercury and its compds are of recognized toxicity. The handling of MF is not unduly hazardous from a toxicity viewpoint, but should be done with the minimum contact with the skin. Its dust should not be inhaled and it has been recommended that, in order to avoid undue toxicity, the air in loading plant buildings should contain not more than 0.1mg of MF per cubic meter (Ref 35)

Trauzl Lead Block Test. See under Power (or Strength)

Vacuum Stability Test at 100°. Explodes (Ref 34)

Velocity of Detonation. See Detonation Rate

Volatility – not found

Uses of MF. Until the invention of LA (Lead Azide), MF was practically the only expl used both in primers, blasting caps and detonators, either by itself or in compns. When loaded alone in blasting caps [also known as "commercial (or nonmilitary) detonators"] it is subdivided into eight numbers accdg to the amt of MF contd in them: No 1 contains 0.30g, No 2 0.40, No 3 0.54, No 4 0.65, No 5 0.80, No 6 1.00, No 7 1.50 and No 8 2.00g. The Table on p 414 of Davis (Ref 23) gives also external dimensions in mm. This classification is international. Nos 6, 7 and 8 caps are the only ones manufd in the US and the No 6 is the one most commonly used (Ref 23, p 416). The fulminate in commercial and military detonators was first modified by mixing it with BkPdr, then with K nitrate, and later with K chlorate. The chlorate mixts soon attained commercial importance and by 1910 had largely displaced straight MF. These were "single component caps" which are described under "BLASTING CAP" in Vol 2 of Encycl, p B185-R. They are known in Gt Britain as "Plain Detonators", as mentioned in Vol 4, p D737, under "Nonmilitary Detonators". Most commonly used mixts have been MF/$KClO_3$–90/10 and 80/10 and weight by weight they are more brisant than straight MF, when determined by Sand Test (See Table on p 417 of Ref 23). These mixts are more hygroscopic than straight MF, but are cheaper and slightly safer to handle and to load. Plain detonators have been largely (but not wholly) displaced during and after WWII by Compound Detonators, which are described as "Compound Caps" in Vol 2, p B185ff

There are in the USA two caps stronger than No 8: 1) US Army Special Blasting Cap, formerly known as "Special Army Engineer Cap" (Vol 2, p B188-R) and "Western Big Inch Cap" (Vol 2, p B186-R). They are "compound caps" but do not contain MF

When MF is mixed with inert materials and/or fuels, such as antimony sulfide (Sb_2S_3), it merely rapidly burns (deflagrates) and can be used (especially if some K chlorate is also included) for igniting propellants, etc (See "Mercuric Fulminate and Its Mixtures and Qualitative & Quantitative Analyses", described after AddnlRefs for SF)

As already mentioned, MF was largely (and now wholly) displaced since 1930 by LA (Lead Azide) and also by DAzDNPh (Diazodinitrophenol), Cyanuric Triazide and LSt (Lead Styphnate). This took place first in

Germany, than elsewhere in Europe and finally in the USA

Specification Requirements and Tests of US Armed Forces are covered by JAN-M-219(1945). It is discussed after AddnlRefs for SF (Silver Fulminate) under "Mercuric Fulminate and Its Mixtures; Qualitative and Quantitative Analyses and Specification Requirements and Tests"

Mercuric Fulminate–Sulfur Compound, $C_2H_4N_2O_2S$; mw 121.14, N 23.13%; crysts, mp – deflagrates below 100°; sol in eth & alc; insol in water; was prepd by heating MF in abs ether & hydrogen sulfide
Refs: 1) Beil **1**, 723 2) Steiner, Ber **8**, 1177(1875); **9**, 779(1876)

Mercuric Pyrofulminate or Pyrofulmin. An expl solid of empirical formula $Hg_6O_5C_8N_{12}$ with Hg content 77.77%; prepd by P.Y. Narayana [Current Sci **13**, 313–15(1944) & CA **39**, 4227 (1945)] by heating MF for 64 hours or until gases CO & CO_2 ceased to evolve. The compd was stable to at least 125°. It is identical with *Pyrofulmin* previously prepd by A. Langhans [SS **17**, 9-11, 18-21 & 26-28(1922) & CA **16**, 2603(1922)]

Narayana presumed that 3 mols of MF polymerized in the manner characteristic of cyanogen compds and then decompd according to equations:

$$3Hg(ONC)_2 \rightarrow Hg_3(O_2N_2C_2)_3 \text{ (hypothetical and unstable)}$$

$$2Hg_3(O_2N_2C_2)_3 \rightarrow Hg_6O_5C_8N_{12} + 3CO_2 + CO$$

Potassium Fulminate, KONO, mw 81.11, N 17.27%; crysts (from alc); very hygroscopic; sp gr 1.80; mp – expl; expl also by the action of flame. Can be prepd by shaking K amalgam with MF either in methanol or abs ethanol. Expln temp in 5 secs 225°
(See Ref 1, p [777] & Ref 23, p 411)

Potassium–Silver Fulminate, $KAg(ONC)_2$, mw 231.01, N 12.13%; mp – expl; sol in 8 parts of w. Can be prepd accdg to Daniel (1902), p 319 by adding KCl to a hot aq soln of SF. When treated with dil nitric acid, a ppt of Acid Potassium Fulminate is formed which is also explosive (See also Ref 1, p 722 & Ref 23, p 413)

Silver Fulminate (Fulminate d'argent in Fr; Knallsilber in Ger; Fulminato d'argento in Ital; Fulminato de plata in Span and Gremucheye Serebro in Russ), AgONO, mw 149.90, N 9.34%; wh crysts or amorphous aggregates; darkens on exposure to light; mp – expl at 170° in 5 sec (Ref 23, p 411); soly in w 0.0075 parts in 100ml at 13°, 0.018 at 30° and 0.25 at 100°; insol in nitric acid and is decompd by HCl
Preparation of SF
Caution: Due to its extreme sensitivity to mechanical action, it should be prepd only in very small quantities. When working in the lab, do it under a hood behind a safety glass or plastic, while for the plant production a room with good ventilation is required; a barricade, and an arrangement for remote control

Accdg to Davis (Ref 23, p 405), E. Howard, after successfully prepg MF in 1799–1800, attempted to prep fulminates of Au, Pt, Sb, Sn, Cu, Fe, Pb, Ni, Bi, Co, As, Mn & Ag, but only with Ag had any success. In 1802, L. Brugnatelli worked out a satisfactory method, which consisted in pouring onto 100 grains of powdered Ag nitrate, first an ounce of alcohol and then an ounce of nitric acid. After the ppt was formed, the slurry was immediately diluted with water (to prevent the dissoln of SF) and immediately filtered

Because SF is extremely sensitive to friction and heat it quickly became an object of amateur interest and public wonderment; one of the standard exhibits of street fakirs, etc. J. von Liebig (1803–1873) saw, when he was a boy, a demonstration of SF in the market place at Darmstadt and learned how

to prepare it. He retained the interest in it and in 1823 conducted studies on SF in the laboratory of J.L. Gay-Lussac (1778–1850) in Paris (Ref 23, p 405)

Laboratory Procedure, as conducted at Picatinny Arsenal and described by S. Livingston was as follows:

1) To 1 g of pure silver powder, placed in a flask, was added a mixture of 8.5g of nitric acid (d 1.42) & 1.2g water preheated to 90–95° and this was left at RT until complete dissoln of silver

Note: All these manipulations were conducted under the hood behind a safety screen

2) The resulting soln of Ag in dilute nitric acid had temp of 60°. It was carefully added to a 150-ml balloon flask provided with a thermometer and contg 12.25g of 95% ethanol (preferrably not denatured)

3) The flask was then placed in a trough which could be filled with either cold or hot w. The temp in the flask was kept below 60°C by cooling the flask, if necessary. If the reaction started to slow down, the flask was slightly heated by adding some hot w to the trough. If some brown fumes started to appear in the flask, the reaction was slowed down by cooling the flask under cold w tap

Note: As the fumes evolved during this operation are very toxic care was taken not to inhale them

4) As the reaction proceeded, all of the SF precipitated in about 20 mins, giving practically 100% yield

For the plant procedure, Livingston recommended using 10–15g of silver and to increase the amounts of other ingredients correspondingly

Note: Essentially the same laboratory procedure was described by Rinkenbach in Kirk & Othmer **8**(1965), p 590. Prepn of SF was also described by Taylor & Burton (See Addnl-Ref C for SF). Prepn of sensitive form of SF was described by Taylor & Rinkenbach (See AddnlRef D for SF)

Explosive Properties of SF. It is a more effective initiating agent than MF, although it has nearly the same brisance, judging by the Sand Test. It is more sensitive to heat than MF judging by its expln temp (170° vs 210° for MF in 5 secs). Its amorphous modification is (accdg to Rinkenbach) less sensitive to impact than its crystalline form. Since it is practically impossible not to have any crysts among the amorphous material, the whole mass might be as sensitive as if it consisted of crysts nearly exclusively

In Table on p 412 of Davis are given the smallest amts, in grams, of SF and MF required to detonate:

	Tetryl	PA	TNT	TNAns	TNX
Silver Fulminate	0.02	0.05	0.095	0.23	0.30
Mercury Fulminate	0.29	0.30	0.36	0.37	0.40

Uses of SF. Extreme sensitiveness, poor stability and high cost prevent the use of SF in commercial or military priming and detonating devices. It has been used however in small quantities in pyrotechnics and in devices used as toys. For example, Daniel (1902), p 318 described petards, called "cosaques", used in France. In the US, Russia and other countries it has been used for prepn of "snaps", "pull-crackers", paper caps for toy pistols and "jumping frogs". Also in amusement devices such as "cigars" and "spiders". If a few grains of wet SF are spread on a floor, loud cracks are heard if they are walked upon (See Beil 1, 722 and AddnlRefs for SF)

Silver Fulminate (SF) and Telescope Mirrors. Telescope mirrors are silvered by prepg a soln of K hydroxide and Ag nitrate ammonia and covering the mirror with it. Unless the prepn steps are followed carefully, SF may form which is very expl. For this reason the USBurStds recommends that goggles be worn to avoid an unnecessary risk. The risk is especially great if the "silvering" soln is kept for more than several hours. SF can expl while wet and it is so sensitive that it detonates by the mere movement of

the vessel contg silvering soln

E.H. Barry of NY told to Mr Cohn of Franklin Institute that, in order to test the stability of silvering soln it was allowed to stand over the weekend in closed room, in an open beaker. On Monday morning the contents and the beaker fragments were found to have been scattered all over the ceiling. A similar accident was described in Scientific American, April 1932

It is recommended, in order to avoid accidents to prep the soln only as required and if any of it will be left over to keep it in tightly corked vessel, to avoid the possibility of lowering the level by evapn and the formation of a dried residue on the side walls. All the vessels used for keeping silvering soln should be carefully cleaned immediately after use

Note: In some labs where BTF worked it was prohibited to pour into the sink any of the silver contg solns. A special bucket was provided for this and the soln was treated in order to recover silver

Refs: 1) A.G. Ingalls, Edit, "Amateur Telescope Making", Book One, 1967, pp 413–15, Scientific American, Inc, NY, 10017, about $8 (Recommended by Mr Ben Harriman, Harrisburg, Pa, 17109) 2) G. Cohn, Edit, Expls&Pyrots **6**(6), 1973 (Abstract of Ref 1)

Sodium Fulminate, NaONC; mw 65.02, N 21.55%; wh crysts, sp gr 1.92, mp – explodes at 215° (in 5 secs) (Ref 23, p 411); sol in w. Can be prepd by shaking Na amalgam with MF either in methanol or absol ethanol. Wöhler et al (Ref 10) prepd several explosive double salts and proposed using some of them in primers and detonators

Davis (Ref 23, p 408) stated that Wöhler detd mol wt of NaF that corresponded to the single monomolecular formula of NaONC. This fact taken, together with the fact that MF warmed with concd aq HCl yields hydroxylamine, NH_2OH, and formic acid proves that Fulminic Acid (qv) is the oxime of carbon monoxide:

$$HO{-}N{=}C\!\!\nearrow\!\!\searrow + 2H_2O \rightarrow HO{-}NH_2 + HCOOH$$

(See Refs 1, 6, 10, 18 & 23)

Sodium Cuprous Fulminate, $Na[Cu(ONC)_2]$, and **Disodium-Cuprous Fulminates,** $Na_2[(ONC)_3].3H_2O$; are solid complexes prepd by L. Wöhler et al and described in Ref 10. The first complex explodes violently by heat or impact. It was prepd by the action of cuprous chloride on NaF in w at 80°. The second complex expl violently on heating. It was prepd by the action of concd soln of MF on cuprous chloride in w at RT (pp 2754–56)

Thallium (Thallous) Fulminate, TlONC; mw 246.41, N 5.68%; colorless solid becoming yel on exposure to light; mp – expl at 120° in 5 secs. Can be prepd by shaking Tl amalgam with MF in ethanol. It is an expl with very low heat of expln – 223cal/g, low gas vol, low brisance, very low sensitiveness and low initiating efficiency. It takes 0.30g of TlF to initiate Tetryl and 0.43 for PA [See Ref 1, p (376); Ref 3, pp 1 & 18 and Ref 23, pp 411–12]

Refs for Fulminates, Including MF:

1) Beil **1**, 720–22, (375), [777] & {2941} 2) L. Wöhler et al, Ber **38**, 1345 & 1351 (1905); Ibid **43**, 754(1910) & **50**(1917) 3) L. Wöhler & F. Martin, SS **12**, pp 1, 18, 39, 54 & 74(1917) 4) R. Escales & A. Stettbacher, "Initialexplosivstoffe", Veit & Co, Leipzig (1917) 5) Marshall **2** (1917), 701 6) A. Langhans, SS **16**, 105 (1921) 7) C.A. Taylor & W.H. Rinkenbach, ArmyOrdn **5**, 463 & 824(1924) 8) Ibid, JFranklInst **204**, 369(1927) 9) Naoúm, Expls (1927), 156 10) L. Wöhler et al, Ber **62**, 2742 & 2748(1929) 11) W.H. Rinkenbach & O.E. Burton, ArmyOrdn **12**, 120(1931) 12) O. Turek, Chimie et Ind **26**, 781(1931) 12a) Ph. Naoúm, SS **27**, 181, 229 & 267(1932) 13) H. Muraour, Bull(Fr), (4), **51**, 1156(1932) 14) W.

Friederich, SS **28**, 115(1933) 15) V.E. Clark, IEC **25**, 663 & 1385(1933) and SS **28**, 345(1933) 16) Stettbacher (1933), p 330 (Knallcadmium), 330 (Knallgold), 330(Knallnatrium), 324–27(Knallquecksilber), 330 (Knallsäure) 17) Pepin Lehalleur(1935) 140–50 18) L. Hackspill & W. Schumacher, AnnAcadSciTech, Varsovie(Poland) **3**, 84 (1936) 19) J.D. Hopper, JFranklInst **225**, 219(1938) 20) R. Wallbaum, SS **34**, 127, 161 & 197(1939) 21) Thorpe **4**(1940), 558; Ibid **5**(1941), 384 22) Clift & Fedoroff, Vol **2**(1943), pp F3 to F5(Fulminates) 23) Davis (1943), 403–14 24) Pérez Ara (1945), 541–52 25) Vivas, Feigenspan & Ladreda, Vol **2**(1946), 306–15 26) F.W. Brown et al, "Sensitivity of Explosives to Initiation by Electrostatic Disc", USBurMines **RI 3852**(1946) 27) Stettbacher (1948), 95–6 28) Belgrano (1952), 200–12 29) Stettbacher, Pólvoras(1952), 124–25 30) Giua, Trattato **6**(1)(1959), 415–18 (Fulminati & 419–22(MF) 31) CondChemDict(1961), 719(MF) 32) Kirk & Othmer, 2nd Edit, Vol **8**(1965), 587–89(MF) & 590 (SF)(by Wm.H. Rinkenbach) 33) Fordham (1966), 103 34) AMCP **706–177**, March 1967, pp 201–05(MF) 35) TM **9–1300–214**, Nov 1967, pp 7-4 to 7-7(MF) 35a) Sax (1968), 784(Dry MF) & 784–85(Fulminates) 36) CondChemDict (1971), 556-L(MF) 37) AMCP **706–177**(1971), pp 201–05(MF) 38) Gorst (1972), 99–100(Gremuchaya rtut' or Ful'minat Rtuti)(MF)

AddnlRefs for MF:.

A) A. Solonina, SS **5**, 41–6(1910)(Prepn of MF) B) O. Hagen, SS **6**, 4–7(1911)(Manuf of MF) C) W.R. Hodkinson, JSCI **37**, 190T (1918)(Props of MF) D) A. Langhans, SS **14**, 300, 318, 334, 350, 366 & 390(1919); Ibid **15**, 7, 28, 219, 227 & 235(1920); Ibid **17**, 122, 131, 141, 150 & 159(1921)l Ibid **18**, 6, 17, 27 & 52(1923) Da) C.A. Taylor & WmH. Rinkenbach, "Explosives", USBurMines Bull **219**(1923), 62 E) M.R. Muller, MAF **4**, Part 4(1925)(Fulminic Acid and MF) (Engl transln by H. Aaronson is in PicArsn Library) F) L.R. Carl, ArmyOrdn **6**, 302–04(1926)(Rates of deton of MF and of its mixts with K chlorate) G) F.D. Miles, JCS **1931**, 2532–42 (Crystn structure of MF) H) Pascal (Russ transln)(1932), p 169 (Prepn of MF and its props) I) S. Livingston, "Nature of Impurities in MF which Give Unusual Explosions", PATR **415**(1933) Ia) H. Muraour & W. Schumacher, CR **198**, 1161(1934)(Combstn of compressed MF in vacuo) J) Pepin Lehalleur(1935), p 141 (Structural formulas proposed for MF); 141–42(Reactions of formation MF from Hg, nitric acid & alcohol; 143–44(Liebig's method of manuf MF); 144–45(Chandelon method of manuf MF); 148–49(Driers for MF) K) J.D. Hopper, PATR **480**(1934)(Study of Pb salts of nitrocompds as substitutes for MF) L) J.D. Hopper, PATR **624**(1935) (Development of detonating compds to replace MF) M) L. Majrich, SS **31**, 147–48 (1936)(MF can be obtd in 99.6% purity by dissolving crude product in a mixt of equal vols of monoethanolamine and ammonia and pptn with dil AcOH) N) Sancho(1941), 188(A brief description of methods for prepg MF used in Spain before WWII that of Bechmann, de Bruyn, Chandelon and Troisdorf Fabrik, DAG)(A list of MF mixts with chlorates, nitrates, sulfur and polvorin used in Span primers and detonators) O) A.F. Belyaev & E.E. Belyaeva, DoklAkadNauk (Russ) **33**, 41–4(1941)(Rate of burning of compressed MF at different pressures) P) C. Swanston, ProcRoySocMed **36**, 633–38 (1943)(Dermatitis caused by MF and Tetryl are discussed) Q) H.S. Mason & I. Botvinik, USPubHealthRept **58**, 1183–86(1943)(Dermatitis caused by MF can be prevented by using special soap contg thioethanolamine and diphenylthiocarbazone) R) Clift & Fedoroff, Vol **1**(1943), Chap XVIII, Part 1, pp 1–4 (Prepn & props of MF and tests) S) Davis (1943), 405–12(Prepn & props of MF) T) Blatt, OSRD **2014**(1944)(Prepn, props & uses of MF) U) E.M. Voroshin & A.I. Borbanev, TrudyKhar'kKhim-TekhnolInst **4**, 145–46(1944) [In manuf of MF the highest

yields, 94–96%, were obtd with 1 p (by wt) of Hg, 8.8p of nitric acid (61.5% strength), 8.8p of 96% ethanol, 8.8p of cp HCl & 1 p of electrolytic Cu] V) P.Y. Narayana, CurrentSci **13**, 313–15 (1944) & CA **39**, 4227 (1945) (Mercuric Parafulminate or Parafulmin) W) Kast-Metz (1944) (Prepn, props and analysis of MF and of its mixtures) Wa) L.R. Carl, JFranklInst **240**, 149–69 (1945) (MF, its catalytic reactions and their relation to detonation) X) All & En Expls (1946), 62–6 (Props & uses of MF); 156 (MF may cause infection from contact with skin cuts) Y) A.F. Belyaev & A.E. Belyaeva, ZhFizKhim **20**, 1381–89 (1946) (Combstn of MF) Z) A.R. Ubbelohde, TransRoySoc **A241**, 286 (1948) (Explanation why MF is less sensitive to grit than LA. Heat of expln of MF 410cal/g vs 364 for LA) AA) E. Ledergerber, SchweizMedWochschr **79**, 263–67 (1949) (Deaths of workers in contact with MF primers was due almost exclusively to the inhalation of vapors) BB) H. Henkin & R. McGill, IEC **44**, 1391–95 (1952) (The lowest expln temp 175° was observed on prolonged heating in modified Marshall App. Heating for 5 secs at 208° produced expln) BBa) Gorst (1957), 108 (Gremuchaya Rtut') (MF) CC) W. Brose, GerP 1016255 (1957) (Prepn of finely crystallized MF of the highest possible bulk density) DD) R.S. Harris, ARDE GB Memo (MX) **42/58** (1958) (Production of MF of improved thermal stability) (Conf – not used as a source of info) EE) M.P. Murgai & A.K. Ray, BritApplPhys **10**, 132 (Mar 1959) (Impact sensitivity of MF) FF) S. Nagayama & V. Mizushima, KôgyôKayakuKyôkaishi **21**, 290–96 (1960) & CA **55**, 24012 (1961) (Crysts of MF of different cryst habits were obtd by varying the rate of crystn from solvents. They differed in expl sensitivity and the crysts from hot w were more sensitive because they did not cont w of crystn)

Addnl Refs for SF (Silver Fulminate):
A) Mellor, "Comprehensive Treatise", Vol 3 (1923), p 381 B) Marshall **2** (1917), 703 (Prepn & props) C) C.A. Taylor & E.P. Buxton, ArmyOrdn **6**, 118–19 (1925) (Prepn & props of SF) D) C.A. Taylor & Wm.H. Rinkenbach, ArmyOrdn **6**, 448 (1926) (Prepn of sensitive form of SF) E) Stettbacher (1933), 328 (Knallsilber) F) Sancho (1941), 193 (Fulminato de plata) G) Davis (1943), 411–14 (Prepn, props & uses of SF) H) Pérez Ara (1945), 551–52 (Fulminato de plata) I) Vivas, Feigenspan & Ladreda, Vol **2** (1946), 315 (Fulminato de plata) J) Stettbacher (1948), 96 (Knallsilber) K) Belgrano (1952), 216 (Fulminato di argento) L) Stettbacher, Pólvoras (1952), 125 (Fulminato de plata) M) J.P. Springer, GerP 953415 (1956) & CA **53**, 10765 (1959) (Uses of SF in pyrotechnic compns) N) Giua, Trattato **6** (1) (1959), 418 (Fulminato d'argento) O) Lange (1961), 676–77 (Props of SF) P) Kirk & Othmer, 2nd Ed, Vol **8** (1965), p 590 (SF) (by Wm.H. Rinkenbach) Q) AMCP 706–177 (1967) – not described R) TM 9–1300–214 (1967) – not described

Mercuric Fulminate and Its Mixtures, Qualitative and Quantitative Analyses and Specification Requirements and Tests

Specification Requirements of JAN-M-219:

1) Moisture

2) Preparation of Sample – Approx 70g of the dry sample are needed to perform the required determinations

3) Appearance – Crystals of sparkling appearance which are white, gray or light gray with a yellowish tint

4) Mercury fulminate – 98.0% (min)

5) Acidity – None

6) Insoluble matter – 2.0% (max)

7) Free mercury – 1.0% (max)

8) Chlorine – 0.05% (max)

9) Sand test – weight of sand crushed, 44g (min)

10) Granulation:

Retained on US Std Sieve No 100, % (max) – 15

Thru US Std Sieve No 200, % (min) – 75

Laboratory Tests

112.1 Moisture

112.1.1 Desiccation Method. Dry to con-

stant weight in a desiccator containing calcium chloride, a known weight 0.4 to 0.7g portion of mercury fulminate. Calculate the loss in weight as the percentage of moisture in the sample

112.1.2 Oven Method. Transfer an accurately weighed portion of approx 1 g of the sample to a tared glass moisture dish. Dry in an oven maintained at 55° to 65° for four hours or to constant weight. Calculate the loss of wt as % moisture

112.2 Preparation of dry sample. Transfer two 35g portions of the sample to two 2½-inch glass funnels, each funnel having a filter paper folded and inserted in it. With the aid of suction, wash the Hg fulminate with enough 95% ethyl alcohol to remove most of the water. Place the filter paper holding the Hg fulminate in a small evaporating dish and dry in a sealed unit electric oven or a steam oven at 40°C–50°C to constant weight

112.3 Appearance. Determine by visual examination

112.4 Mercury Fulminate. Transfer a weighed portion of 0.3g of the dried sample to a 250ml beaker. Add quickly 30ml of a freshly prepd soln of sodium thiosulfate (20g per 100ml). Shake the mixture for exactly 1 minute. Titrate immediately with approx 0.10N HCl using three drops of methyl red indicator. It is important that titration begin exactly 1 minute after the addition of the sodium thiosulfate.

Add most of the HCl required immediately and complete the titration in not more than 1 minute. Make a blank determination with the same quantity of sodium thiosulfate solution. Calculate the percentage of Hg fulminate as follows:

$$\% \text{ Hg fulminate} = \frac{7.1116(V-v)N}{W}$$

where: V = ml HCl required for titration
v = ml HCl required for blank
N = normality of HCl used
W = weight of sample

112.5 Acidity. Transfer a weighed portion of 10.0±0.1g of the dried sample to a filtering crucible provided with a filter paper disc. Wash the sample with two 25-ml portions of boiled distilled water which has been cooled to RT. Add to the filtrate three drops of methyl orange or bromthymol blue indicator. Note if acid color appears

112.6 Insoluble matter. Transfer to a 2-liter beaker a weighed portion of approx 1 g of the sample. Add 1 liter of distilled water at 90°C and stir while maintaining at a temperature of 90°C, until the material is dissolved. Filter the soln and transfer any insol material to a tared glass filter or crucible. Complete the soln and filtration within 1 hr. Prepare the filter or crucible by packing fine asbestos to a thickness of approx 0.2 inch on the sintered glass plate of medium porosity, washing with hot water, alcohol and ether while applying suction, and then weighing. Wash the insoluble material on the filter with hot water, alcohol & ether, and weigh. Calculate the increase in wt as % insoluble matter

112.7 Free Mercury. To the insoluble matter, obtained as described in paragraph 112.6, add 2ml of hot concentrated HCl, allowing this to remain for 1 minute prior to removal by suction. Subject the insoluble matter to this treatment three times, wash with hot water, alcohol & ether, and weigh. Repeat the procedure until the loss in weight is less than 0.0003g. To the residue add three successive 2-ml portions of hot dilute nitric acid (1:1) in the same manner as with the HCl. Wash the residue with hot water, alcohol & ether, and weigh. Calculate the loss in wt as % of metallic mercury

112.8 Chlorine. Transfer a weighed portion of approx 5g of the dried sample to a filtering crucible equipped with a filter paper disc. Wash with two 25-ml portions of distilled water at 90°–100°C, catching the filtrate in a test tube. Add to the filtrate three drops of nitric acid (sp gr 1.4) and 10 drops of a 10% soln of silver nitrate. Compare the turbidity with that produced when a soln of 0.0042g of pure NaCl in 50ml of distd water is treated in the same manner. A greater turbidity indicates the presence of more than 0.05% chlorine in the sample

112.9 Sand Test. Transfer to each of five empty No 6 blasting cap shells 0.400g of trinitrotoluene (grade I, US Army Specification 50–13–5) of such fineness that all of the material passes thru a 177 micron (No 80) US Std

sieve. Each blasting cap shall be composed of copper, gilding metal, or aluminum and shall be approx 1.46 inches long by 0.217 inch inside diam. With the cap held in a loading block, insert a plunger 0.20 inch in diam in the shell and subject the TNT to a pressure of 3000psi (100 lbs actual load) for 3 mins. Remove the plunger and transfer to the shell 0.270g of Hg fulminate, insert a reinforcement cap (approx 0.217 inch outside diam, open at one end and a hole 0.11 ± 0.03 inch diam at the other end) with the open end down. Insert the plunger in the shell and apply the pressure of 3000psi for 3 mins. With a pin, prick the powder train in one end of a piece of miner's fuse 8 or 9 inches long. Crimp to the pricked end one of the caps loaded as directed above, taking care that the end of the fuse is held firmly against the charge in the cap. Crimp near the mouth of the cap to avoid squeezing the charge. Pour 80.0 ± 0.1g of standard Ottawa sand which passes thru a 840 micron (No 20) US Std sieve and is retained on a 590-micron (No 30) US Std sieve, into the cavity of the sand test bomb. Level the sand by striking the bomb vigorously with a hammer at least five times while rotating the bomb on its axis. Lower the fuse and cap into the bomb cavity so that the cap is centered at the axis of the bomb and just touches the sand. Hold the cap and fuse in this position and carefully pour 120.0 ± 0.1g more sand into the bomb, being careful not to disturb the position of the cap. To avoid possible error caused by the explosion blowing crushed sand thru the hole in the cover, slip a piece of rubber tubing, approx 1/8 inch long and of such inner diam that it fits the fuse snugly over the fuse and adjust at a point on the fuse so that the rubber will be against the inner side of the bomb cover with the loaded cap in position as described above. Carefully insert the fuse thru the hole in the bomb cover, lower the cover into position, and fasten securely. Strike the bomb vigorously with a hammer at least 5 times while rotating the bomb on its axis. Light the fuse, and after the explosion takes place, transfer the sand to a sheet of smooth glazed paper, taking care to remove any sand which may adhere to the sides of the bomb, or to pieces of the detonator shell, or burnt fuse. Empty all the sand on a 590 micron (No 30) US Std sieve fitted with a pan. Weigh the sand which passes thru the sieve after shaking 3 minutes, using a mechanical shaker

112.10 Granulation

112.10.1 Wet Method. Transfer a dried and weighed portion of approx 10g of the sample to a 149-micron (No 100) US Std sieve which has been placed in a porcelain evaporating dish containing sufficient water to cover the wire portion of the sieve. Shake the sieve for 10 mins in such a manner that the Hg fulminate is below the surface of the water and any material passing thru the sieve is caught on the evaporating dish. Transfer the portion of the fulminate retained on the sieve to a tared filter paper and wash twice with alcohol and once with ether. Dry the filter paper and fulminate at 50°C for 1 hr, cool in a desiccator, and weigh. Calculate the increase in wt as % retained on the No 100 sieve. Transfer the portion of the sample caught in the evaporating dish to a 74-micron (No 200) US Std sieve and sift as described above. Transfer the material caught in the evaporating dish to a tared filter paper, wash, dry and weigh as before. Calculate the increase in wt of the tared filter paper as % passing thru the No 200 sieve

112.10.2 Dry Method. When Hg fulminate is sifted dry, the operation shall be performed in a location safely removed and/or shielded from other explosives, personnel or material which should be harmed by explosions. The motor of the shaking machine shall be started and stopped by a remote control switch. Assemble a No 100 sieve on a No 200 sieve and fit them on a pan. The sieves must be fitted with rubber gaskets. Place a 5g sample of dry fulminate on the upper sieve and shake the sieve for 3 mins on a Tyler Ro-Tap (or equal) machine. Carefully brush the material retained on the upper sieve into a weighing pan and weigh. Calculate the wt as % retained on the No 100 sieve. Brush the material passing thru the No 200 sieve from the pan, weigh & calculate as % passing a No 200 sieve

Methods of detection and determination of MF are listed in Refs which follow

The following mixes contg MF used in fuzes are described in Vol 4 of Encycl:
p D1060-R. *Primer Mix, New No 4* – formerly used in rifle grenade fuzes and covered by the US Military Spec **MIL-P-2499**(1950). The Spec is now cancelled
p D1063-R. *Primer Mix for M3 Igniter Cartridge* used in 81mm Mortar Ammunition contains MF, Sb_2S_3, ground glass, $KClO_3$ and water-soluble binder. Its requirements and tests are covered by US Military Spec **MIL-C-20480A** (1954)

Compns of some US Priming Mixes contg MF and used in fuzes are given in **TM 9-1300-214** (1967), p 7-17, entitled "Military Explosives"

Priming Compns Contg MF

	I	II	III
K chlorate	45	35	14
Sb sulfide	23	30	21
MF	32	35	28
Ground glass	–	–	35
Shellac	–	–	2

I is loaded dry but can be wet loaded with alc
II is loaded dry
III is prepd with shellac dissolved in alc and wet loaded

Refs for Analysis of MF and Its Mixtures:
1) H.W. Brownsdon, JSCI **24**, 381–82(1905) (Quantitative detn of MF); also described and reviewed by R. Philip, SS **7**, 109ff(1912) 2) A. Solonina, SS **5**, 41–6(1910)(Detn of MF) 3) Marshall **2**(1917), 701–02(Estimation of MF, including Bofors Method) 4) G.S. Heaven, JSCI **37**, 143T(1918)(Analysis of MF) 5) A. Langhans, ZAngewChem **31**(I), 161–3 (1918) & JSCI **37**, 637A(1918)(Colorimetric detn of MF with Phenylhydrazine) 6) P. Nicolardot & J. Boudet, CR **166**, 288–89(1918) (Analysis of MF) 7) H. Rathsburg, Ber **54B**, 3185(1921)(Analysis of MF) 8) A. Langhans, ZAnalChem **60**, 93–4(1921)(Detection of MF) 9) A. Langhans, SS **17**, 122ff(1922) & **18**, 6ff (1923) 10) C.A. Taylor & Wm.H. Rinkenbach, "Explosives", USBurMinesBull **219**(1923), pp 62–3(Analysis of MF); 162(Analysis of fulminate–chlorate–Tetryl detonating mixts); 163(Analysis of fulminate–chlorate–TNT detonating mixts); 163(Analysis of fulminate–chlorate–NMan–NC detong mixts); 164(Analysis of MF–NC initiator); 164(Analysis of a loose MF–chlorate initiator); 167–68(Analysis of primer mixts of MF, chlorate and antimony sulfide); 168(Analysis of primer mixt: MF, K chlorate, antimony sulfide & glass); 171(Analysis of primer mixt: MF, Pb Picrate, Sb sulfide, Ba nitrate & Ba carbonate); 172(Analysis of primer mixt: MF, Sb sulfide, Ba nitrate, Picric Acid and glass) 11) Clift & Fedoroff, Vol **2** (1943), pp F4 & F5(Bofors Method of Estimation of MF content); p F5 (Color reaction of MF with Phenylhydrazine) 11a) Kast-Metz (1944)(Testing of MF) 13) Belgrano(1952), 213–6(Analysis of MF and its mixts) 14) H.C. Sayce, ERDE GB Report No **33/R/59**, Jan 1960(Analysis of MF and its application to Fulminate in storage)(Conf – not used) 15) Anon, "Analytical Methods for Powder and Explosives", AB Bofors-Nobelkrut, Bofors, Sweden(1960), 215–17(Analysis of MF) 16) A.R. Lusardi, "Laboratory Manual", FREL Picatinny Arsenal, Sections 100 & 200(1962), Method 112(MF, applicable Spec JAN-M-219); Method 203(Analysis of Primer Mix New No 4, which contains MF, K chlorate & Sb sulfide; applicable Spec MIL-P-2499) 17) "Military Standard Explosive: Sampling, Inspection and Testing", **MIL-STD-650**(3 Aug, 1962), Method 410.1(Detn ofMF content in primers or the percent purity) 18) StdMethodsChemAnalysis, Vol **2**, Pt B(1963), Chap 32 by R.H. Pierson; p 1346(Detection of MF); p 1366(Analysis of compn contg MF & K chlorate) 19) J. Hetman, Explosivstoffe **12**(1964)(Testing of MF by means of cathode-ray polarograph) 20) US Military Specification JAN-N-219(1945)(Mercury Fulminate) 21) US Military Specification MIL-P-2499(Oct 1950)(Analysis of Primer Mix New No 4, contg MF, K chlorate & Sb sulfide)

Fulminatine. An old Dynamite proposed by Mr Fuchs of Upper Berau, Silesia. It consisted of NG–68% or more adsorbed on "laine tontisse" (sheared wool)
Refs: 1) Daniel(1902), 349 2) Gody(1907), 359

Fulminating. An older expression meaning capable of causing explosion or detonation. It is derived from Latin *fulmen*, which means lightning flash or thunderbolt
Refs: 1) Davis (1943), p 400 2) Hackh's Dict (1944), p 359-R

Fulminating Compounds. Fulminating gold, mercury, silver and platinum are formed by precipitating solns of these metals with ammonia. They are perhaps nitrides or hydrated nitrides, or perhaps they contain hydrogen as well as nitrogen and water of composition, but they contain no carbon and *should not be confused with fulminates* which are salts of Fulminic Acid, HONC. They are dangerously sensitive, and not suited for practical use

Their discovery is described under individual compounds
Refs: 1) Davis (1943), 400 2) Clift & Fedoroff **2**(1943), p F6

Fulminating Gold (FG) (L'or fulminant in Fr, Aurum fulminans in Latin, Knallgold in Ger, Oro fulminante in Ital or Span, Gremucheye zoloto, in Russ). This compd and other *fulminating* compds must not be confused with *fulminates*, described above. Some scientists, accdg to Daniel (Ref 1, 314) consider them identical with *nitrides* (azotures, in Fr)

Accdg to Davis (Ref 4, p 400), FG was discovered by Basil Valentin (German monk and alchemist living in the 15th century), but the first known description of this discovery is given in writings of Johann Thölde (or Thölden), published in 1602–1604. The compd was called *Goldkalk* and was prepd by dissolving gold in *aqua regia*(which was made by dissolving *sal ammoniac*, NH_4Cl, in concd nitric acid) and then adding K carbonate soln. The resulting powdery ppt was washed by decantation 8–10 times, drained from water and then dried in the air where no sunlight fell on it and not near any fire. The compd was very sensitive to heat and could explode with great violence. The author also reported that warm vinegar converted the expl compd to an inexplosive material

The name *aurum fulminans* was given by Beguinus, who described in 1608 its prepn (Ref 4, p 401)

Daniel (Ref 1) stated that *l'or fulminant* deposits as a chamois (clear yel) powder on treating gold trichloride with ammonia. The same method is listed by Davis on p 401, who also says that the method of prepn by Basil Valentin succeeded because the sal ammoniac used for the prepn of the aqua regia supplied the necessary ammonia. If gold is dissolved in aqua regia prepd from nitric acid and common salt, and if the soln is treated with K carbonate, the resulting ppt is not expl. FG loses its expl props if it is allowed to stand in contact with sulfur

Stettbacher (Ref 3) stated that Aurum fulminans can be prepd by treating auric oxide, Au_2O_3, (freshly pptd by treating the soln of auric chloride with an alkali and filtering) with ammonium hydroxide in the manner similar to prepn of Fulminating Silver (qv). It forms olive-green to yel-brn powder which in dry state detonates with sharp sound on being touched

Accdg to Stettbacher, the constitution of FG was not defined, but accdg to Pérez Ara (Ref 4, p 567), the formula of *oro fulminante* is $Au(N_3)_3$ and that it is *nitruro aurico* (Gold Nitride in Engl). This formula, however, seems to be identical with an azide

Stettbacher, quoting the work of Langhans (Ref 2), stated that Aurum fulminans is the most ancient *brisant explosive* known and that manipulation with it in the past caused many serious accidents. For example, the famous Swedish chemist Baron J.J. Berzelius (1779–1848) lost both eyes while working with FG (Ref 2, p 330)

Accdg to Davis (Ref 4, p 401) and Pérez Ara (Ref 4, p 567), Cornelius Drebbel, the Dutch inventor and chemist of the 17th century, while being in the service of British Navy, devoted considerable time to the prepn of FG and used his material as a detonator in petards and torpedoes used in the English expedition of 1628 against the French port of La Rochelle

Accdg to the diary of Pepys of Nov 11, 1663(quoting from Ref 4, p 401), FG is so powerful and brisant that if one grain of it is

exploded on a surface of a silver spoon, a blow like a musket was heard and this punched a hole thru the spoon

FG is not suitable for practical use on acct of its extreme sensitiveness to shock, friction or heat, and also to its high cost (Ref 5, p 568

(Compare this compd with *Auric Imidoamide* or *Gold Amidoimide*, described in Encycl under AMIDES, IMIDES AND DERIVATIVES on p A169-L of Vol 1. It seems that the compd there was erroneously named Fulminating Gold)

Refs: 1) Daniel (1902), 313–14 2) A. Langhans, SS **25**, Sonderdruck 70 (1930) (Explosionen, die man nich erwarten) 3) Stettbacher (1933), 330–31 4) Davis (1943), 400–01 5) Pérez Ara (1945), 567–68

Fulminating Mercury or **Mercury Nitride.** Accdg to Marshall (Ref 1), its formula is Hg_3N_2, mw 629.85, N 4.45% and it can be prepd by treating mercury oxide with ammonia. It is very sensitive and must not be confused with Mercury Fulminate

Accdg to Davis (Ref 2), Fourcroy, by digesting red oxide of mercury (HgO) in ammonia water for 8 to 10 days, prepd a material which became white and finally assumed the form of crystalline scales. The dried product exploded loudly from fire, but underwent spontaneous decompn when left alone. At slightly elevated temp it gave off ammonia and left a residue of mercury oxide

In the Journal de Physique for 1779 the French apothecary Bayen described a fulminating mercurial compd of a different kind. It was obtd by mixing 30 parts of precipitated Hg oxide (washed and dried) with 4 or 5 parts of sulfur. This mixt exploded violently when struck with a heavy hammer or on being heated on an iron plate

Accdg to Pérez Ara (Ref 3), there exist two *nitruros de mercurio* (Mercury Nitrides) – one is mercurous, while another is mercuric

None of the above listed expls found application in commercial or military primary or detonating compns, but they are interesting from an historical point of view

Refs: 1) Marshall **2** (1917), 703 2) Davis (1943), 402 3) Pérez Ara (1945), 567

Fulminating Platinum (FP) (Platine fulminant in Fr)

Accdg to Daniel (Ref 1), when a soln of platinic oxide in sulfuric acid was treated with an excess of aq ammonia; a black ppt of platine fulminant was obtd. When dry it detonated violently by shock, friction and heat (at 160°). Some scientists consider it as being identical with the *nitride* (azoture, in Fr)

Accdg to Davis (Ref 4, p 402), FP was first prepd by Davy (Sir Humphry, 1778 to 1829) about 1825, by adding ammonia water to a soln of Pt sulfate, boiling the ppt with a soln of potash, washing, and allowing to dry. It was exploded by heat, but not easily by percussion or friction. No formula is given

Accdg to Jacobson (Refs 2 & 3), the compd of the formula $(HO)_5PtNH_3Pt(OH)_5$ was prepd by him by treating dichloroplatinic acid with an excess of ammonia. The soln first turned dark and then a brownish ppt appeared, which could be separated and dried. When heated, it blackened and detonated violently yielding Pt, N_2, O_2 and H_2O vapor. Dichloroplatinic acid was obtd in 80–90% yield by boiling Ag tetrachloroplatinate in water

A compd of similar props, $(HO)_5PtC_5H_5NPt(OH)_5$, was obtd by the action of pyridine on dichloroplatinic acid

Refs: 1) Daniel (1902), 313 & 314 (Platine fulminant) 2) J. Jacobsen, CR **149**, 574–77 (1909) & CA **4**, 1002 (1910) 3) Clift & Fedoroff **2** (1943), p F6 4) Davis (1943), 402

Fulminating Powder of Forsyth. Under this name Newman (Ref) lists the mixture of K chlorate, sulfur & charcoal which could be detonated by percussion. It was invented at the beginning of the 19th century by the Scotch clergyman, A. Forsyth. It was replaced later by the mixture consisting of K chlorate, MF & powdered glass

Ref: J.R. Newman, "Tools of War", Doubleday-Doran & Co, NY (1943), p 42

Fulminating Powders Described by Davis. One of the *fulminating powders* was made, accdg to Ure's "Dictionary of Chemistry", Philadelphia, Pa, 1821, by triturating in a warm mortar 3 parts by wt of nitre, two of carbonate of potash and one of flower of sulfur. Its effects, when fused in a ladle and then set on fire, were very great. The whole of the melted material exploded with an intolerable noise, and the ladle disfigured, as if it had received a strong blow downwards. It was three times quicker than common BkPdr

A similar powder was manufd by S. Guthrie, Jr and sold in 1930's in this country. He proposed, however, another powder, which he claimed to be superior to the above mixture. For its prepn were melted together 2 pts of nitre with 1 pt of K carbonate and, when the mass cooled, adding to 4½ ps of it 1 pt of sulfur. This pdr was claimed to be no less than 8½ times quicker than the best common BkPdr. More details are in Ref

Ref: Davis (1943), 31

Fulminating Powders, History. It seems that the earliest discovery dates to the 15th century, when *Fulminating Gold* (qv) was prepd by Basil Valentin. Discoverers of other *fulminating compds* are listed under individual compds

A *résumé of description given in the book* of Greener (Ref) is given below:

Accdg to him the first reséarchs on *fulminating powders* were made in France by Peter Bolduc prior to 1700 and also by Nicolas Lemery prior to 1712. Nothing of importance appears to come out of their researchs, but the work was continued in that direction until 1774 when Bayen (Chief Army Physician to Louis XV) prepd MF and made known its expl props. No use of it was made at that time, due probably to its extreme sensitiveness. The work on fulminating expls was continued by Fourcroy, Vauquelin and Berthollet (qv). The last scientist discovered K chlorate and proposed using it in lieu of K or Na nitrates in BkPdr. The resulting *Berthollet Powder* (qv) was too dangerous to handle (See Vol 2 of Encycl, p B107-L). Then Berthollet turned his attention to fulminate and succeeded in preparing *Silver Fulminate* (qv) and used it for pyrotechnic displays. Attempts to use it as a primer in firearms were unsuccessful due to its even greater sensitiveness than that of MF. Fourcroy & Vauquelin attempted to diminish the sensitiveness of SF by combining it with combustible ingredients, such as sulfur, but it does not seem that they succeeded Same investigators tried other fulminating mixts, such as Ag chlorate with sulfur; K iodate with Ag powder and Au, K or Ag Ammoniates (See AMMINE or AMMONIATE in Vol 1 of Encycl, pp A275ff)

In 1800 E. Howard, an Englishman, after a study of works of French chemists, prepd a mixture consisting of MF and saltpeter. This mixt, known as *Howard's Powder* was suitable for use in primers

None of the above-mentioned scientists (including Howard) patented his inventions and for this reason the invention of fulminating mixture is generally attributed to the Scotch Clergyman, A.J. Forsyth, who took in 1809 a patent relating to the application of the detonating principle for exploding BkPdr in firearms (See also "Fulminating Powder of Forsyth")

A short time afterwards (1808), a Swiss gunmaker, Pauly, working in Paris, invented a percussion breech-loading gun in which a fulminating paper cap was affixed to the cartridge in the breech. Upon pulling the trigger a needle pierced the cap which ignited the charge of fulminating mixture. Although this invention was soon abandoned, Germans used the same idea when they patented in 1836 their *needle gun*

In 1812, Pauly invented another percussion device. Here, a small piston, actuated by a spiral spring, struck a nipple upon which a few grains of MF were placed

In 1818, the copper percussion caps were made in England, simultaneously by several gunsmiths, among them Joe Manton. Col Hawker claimed that he improved the Manton cap. In France, the copper cap was introduced by Degoubert in 1819

Among other English inventors of percussion devices may be mentioned Egg, Wilkinson, Lancaster, Long and Westley-Richards

Notwithstanding the advantages of percussion devices over the *flintlock* (qv), they were not adopted until 1840 by the English and French

Refs: 1) W.W. Greener, "The Gun and Its Development", Cassell, Petter, Galpin & Co, London (1885), pp 93–100 2) Ditto, 9th Edition (1910) (A Facsimile Reprint obtd from Publisher's Central Bureau, Long Island City, NY 11101)

Fulminating Silver of Berthollet. (L'argent fulminant de Berthollet in Fr; Berthollet'schen Knallsilber in Ger). This compound, of indefinite formula, was first prepared in 1667 by Baron Johann Kunckel von Löwenstern (See under Mercury Fulminate) and then, more than one hundred years later, by Berthollet (Ref 1). Other chemists, such as Gay-Lussac, Faraday, Proust, Higgins, Raschig, Matignon etc, were interested in the compd and many accidental explns occurred during its prepn and investigation (Ref 4a)

Berthollet (Refs 1 & 7) prepd FS in 1788 by adding lime water to a soln of Ag nitrate, separating the resulting ppt of Ag oxide, drying it and treating with strong Amm hydroxide. The resulting black solid was washed by decantation and dried on filter in open air. It could also be prepd by adding ammonia to very small quantities of the aq soln of Ag nitrate until the ppt, formed at first, is redissolved; NaOH was then added which pptd a black "fulminating silver". It was a very violent expl of indefinite compn (Ref 5). Several formulas were ascribed to this compound, such as $AgNH_2$, Ag_2NH, Ag_3N_2, Ag_3N, $AgO.2NH_3$ etc, but according to the investigation of Baum (Ref 4), its formula is $Ag_2O.NH_3$

Accdg to Davis (Ref 7, pp 401–02), FS is more sensitive to shock and friction than FG, being exploded when touched. It must not be enclosed in a bottle or transferred from place to place, but must be left in the reaction vessel, or better upon paper, where it is allowed to dry

Note: The black material which deposits in a reagent bottle of ammoniacal Ag and sometimes collecting on the rim & around the stopper contains FS. Explns have been reported being caused by careless turning of the glass stopper of a bottle contg this reagent. After a test (for aldehyde, for example) has been made with ammoniacal Ag nitrate soln, the liquid should promptly be washed down the sink and all insol matter left in the reaction vessel should be dissolved with nitric acid

It is too sensitive to be used for any practical purpose (See also Silver Amide, under Amides, Inorganic in Vol 1 of Encycl, p A169-R)

Refs: 1) C.L. Berthollet, AnnChimPhys 1(1), 52(1789) 2) Marshall 2(1917), 703 3) J.W. Mellor, "A Comprehensive Treatise" Vol 3, 380–81(1923) 4) F. Baum, ChemZtg 53, 354 & 374(1929) & CA 23, 4113(1929) 4a) A. Langhans, SS 25, 70(1930) (Explosions which are not expected) 5) P. van der Willigen, ChemWeekblad 29, 232(1932) & CA 26, 6140(1932) 6) Stettbacher(1933), 330 7) Davis(1943), 400–02

Fulminatin (Ger). Mixture of NG 85 & wool shearings (clippings) 15% proposed by Fuchs of Alt Berau, Silesia

Refs: 1) Daniel (1902), 319 2) Gody (1907), 359 3) PATR 2510(1958), p Ger 53-L

Fulminic Acid or **Paracyanic Acid** (Oxime of Carbon Monoxide) (Knallsäure in Ger; Acide fulminique in Fr; Gremuchaya Kislota in Russ), HONC, mw 43.03, N 32.56%, OB to CO_2 −55.8%. Known in solution, forms variously expl metallic salts known as fulminates. The mercury salt, apparently the first discovered was formed by the action of mercuric nitrate on ethyl alcohol (Ref 2). The free acid can be isolated in cold ether and below −5° may exist as a gas for a short time. When heated it polymerizes to meta-fulminuric acid, HC.C(=NOH)C(=NOH)O.N. Other methods of prepn are boiling an aqueous soln (with or w/o mineral acid) of methyl nitrolic acid, $O_2N.CH=NOH$, and then adding a metal salt to precipitate the fulminate (Ref 3) or acidifying aminomethylnitrolic acid, $O_2N.C(NH_2)=NOH$, aminoformoxime, $H_2NCH=NOH$ (Ref 4). Davis

(Ref 5, p 408) shows why fulminic acid has monomolecular formula (See here under Sodium Fulminate)
Refs: 1) Beil **1**, 720 2) E. Howard, PhilTransRoySoc **1800**, 204 3) H. Wieland, Ber **40**, 418–22(1907) & CA **1**, 1270–1(1907) 4) Ibid **42**, 820–2(1909) & CA **3**, 1271(1909) 4a) Pepin Lehalleur (1935), 140(Gives formula C≡NOH and lists four other formulas, which he considers unsuitable) 5) Davis (1943), 400, 407, 408 6) F.C. Palazzo, GazzChimItal **79**, 3–13(1949) & CA **44**, 2929h(1950)(Review on polymerization)

Fulmipaille or **Paléine.** Dynamite invented by Langfrey in 1878, which contained: Fulmipaille (Nitrostraw) 18.6, NG 35.0, saltpeter 32.5, flowers of sulfur 4.6 & starch 9.3%. The last two ingredients can be replaced by charcoal and by dextrine
Ref: Daniel, Dict(1902), 319 & 596

Fulminuric Acid (Nitromalonsäure-amid-nitril in Ger) and **Fulminurates,** $NCCH(NO_2)CONH_2$; mw 129.08, N 32.56%, OB −55.8%; prisms(alc), mp 145°(dec); sol in w & alc. Is tautomeric with $NCC(=NOOH)CONH_2$. Obtd by spontaneous dec of $O_2NCH(CH=NOH)_2$ in water (Ref 3); by treating with concd ammonia $NCC(NO_2K)CO_2Et$ (Ref 4); by refluxing Mercury Fulminate with aq NH_4Cl (Ref 2); to obtn the free acid from its alkali salts, convert to the Ag salt with $AgNO_3$ and treat with HCl or H_2S or convert to the basic Pb salt with basic Pb acetate and treat with H_2S

Following are its salts:
Ammonium salt, NH_4("F"), prisms; sol in hot w (Ref 2)
Copper salt, Cu("F")$_2$.$4H_2O$, rh green crysts (Ref 1)
Copper salt, Cu("F")$_2$.$4NH_3$, dk bl prisms, expl over 150°. Prepd by refluxing fulminuric acid with ammoniacal copper soln (Ref 1)
Mercury salt, Hg("F")$_2$, cryst powd. Prepd from fulminuric acid and $Hg(NO_3)_2$; also a *monohydrate* prepd using HgO(Ref 1)
Potassium salt, K"F", prisms; sol in cold w, dec over 225° with expln at higher temp (Ref 2)
Silver salt, Ag"F", ndls; sol in hot w (Ref 2)
Refs: 1) Beil **2**, 598 2) J. Liebig, Ann **95**, 283–9(1856) 3) H.H. Hill & W.J. Hale, AmChemJour **29**, 260(1903) 4) M. Conrad & A. Schulze, Ber **42**, 740(1909)

Fulmison. A Dynamite containing nitrated bran with 30–40% of NG
Ref: Daniel (1902), 320

Fulmitriguanurate, $C_6H_{11}N_9O_3$; mw 257.22, N 49.01%; wh ndls, sol in warm w. Structure not known. Prepd by heating Mercury Fulminate with ammonia at 70°. Completely decomposed to CO_2 and NH_3 by dil HCl at 150°. Neutral in water, but will react with metal oxides giving, eg, a *Silver salt,* $Ag_2C_6H_9N_9O_3$
Refs: 1) Beil **1**, 723 2) A. Steiner, Ber **8**, 522(1875) 3) Ibid, **9**, 784(1876)

Fulöph & Lackovic Explosive (Hungarian): Fresh horse dung 60, K nitrate 26, sulfur 10 & dye 4%
Note: Accdg to Daniel (Ref 1), fresh horse dung contains ammoniacal salts, which can form an explosive when mixed with other ingredients
Refs: 1) Daniel(1902), 320 2) Fedoroff & Clift **4**(1946), 37

Fumaric Acid and Derivatives
Fumaric Acid or *Butanedionic Acid,* HOOC.CH:CH.COOH; mw 116.07, colorless prisms, mp 286–87°(in sealed tube), bp 290°; sl sol in w & in eth; sol in alc. Can be prepd by fermentation of molasses; isomerization of maleic acid or catalytic oxidation of benz. Used for prepn of polyester resins, molding powders and for many other purposes indicated in Ref 2
Refs: 1) Beil **2**, 737, (299), [631] & {1890} 2) CondChemDict (1961), 518-L; (1971), 404-L

Fumaric Acid Diazide or **Fumaryl Azide,** $(N_3)OC.CH:CH.CO(N_3)$; mw 166.10, N 50.60%, OB to CO_2 −67.5%; fine crysts, explodes vio-

lently at 45° even when heated slowly; very sensitive to impact while wet; can be prepd by introducing $NaNO_2$ in water to an aq soln of fumaric hydrazide acidified with acetic acid. The product is too sensitive for practical use

Refs: 1) Beil **2**, not found 2) H. Ficheroulle & A. Kovache, MP **41**, 12–13(1959) 3) W.G. Rose & H.P. Lundgren, USP 2881046(1959) & CA **53**, 14535(1959)

Fumaric Acid, Nitrated Derivatives, not found in Beil or in CA thru 1966

Fumaric Acid Peroxide or **Fumaryl Peroxide** (Polymers), $[O.OC.CH{:}CH.CO.O]_x$ mw 114.06_x, O 56.11%; wh, very unstable powder; mp – puffs off at 80°; insol in w & alc. Can be prepd by treating fumaric acid dichloride with Na peroxide in ice water. Explodes on heating or when brought in contact with aniline or alcoholic ammonia

Refs: 1) Beil **2**, 743 & [630] 2) L. Vanino & A. Thiele, Ber **29**, 1726(1896) 3) F. Fichter & A. Fritsch, Helv **6**, 333(1923) and JCS **124**, I, 438(1923)(Decompn by heat)

Fume Characteristics. In commercial blasting, one needs to know what poisonous gases & condensed products may be produced and in what proportions they may be found in the atmosphere following a blast. The poisonous gases are referred to in the expl industry as *fumes*. Limitations of the various methods used to measure fumes (Bichel gauge, Crawshaw gage & Trauzl block) have been recognized for years. The USBurMines classification of permissibles into Class A & Class B is based on a closed-bomb method

Many studies more nearly representative of field conditions have been carried out by various investigators. One important finding was the observation of appreciable undetonated expl or expl ingredients in the products of deton. Nitrostarch expls have fume characteristics somewhat better than comparable grades of NG Dynamites, but they do not possess many other desirable props of dynamites

Ref: Cook(1958), pp 15 & 283

Fumée. Fr for smoke

Fumée, Poudres sans. Fr for Smokeless Propellants

Fumelessite. A smokeless proplnt invented by Boyd of Birmingham in 1896. Its compn was similar to that listed under Boyd Powders in Vol **2** of Encycl, p B259-L

Fume-Off. The oxidative destruction of by-products in nitration reactions

Ref: Coll, "Summary Report of Div 8, NDRC", Vol **1**(1946), p 153

Fume Recovery at US Plants Manufacturing Nitrocellulose. Manuf of NC is described in Vol **2** of Encycl, p C102-R, under CELLULOSE and in Vol **3**, pp C536 to C539, under CORDITE. However, no "Fume Recovery" was described although we had in our possession sketches with brief descriptions of fume recovery methods as practiced during WWII at Radford Ordnance Works, Virginia, operated by the Hercules Powder Works and at Indiana Ordnance Works, Charlestown, operated by the DuPont & Co

For addnl info, see

Ref: 1) W. de Sveschnikoff, USP 1406353 (1922) & CA **16**, 1321(1922)

FUME TESTS

(Nitrocellulose Stability Tests)

Several NC stability tests are based on the direct observation of the appearance of brown fumes produced on heating NC

Simon Thomas Fume Test is one of the oldest (1898) of its kind and was conducted by heating an expl in a loosely stoppered flask to 100° for eight hours each day until

the appearance of brown fumes (Refs 1, 10 & 11). The same test was used in Holland in a slightly modified form (Ref 5), as follows:

A 2.5g sample of air-dried NC was placed in a 30ml flask and heated, unstoppered, for four hours at 95°

The flask was left until the next day, then it was stoppered and heated for 8¼ hours

This was repeated for as many days as required to produce brown fumes, distinctly visible when the flask was held against a white background

Generally, NC of 11.0–11.5% N required about 24 days; 11.5–12.0% – 23 days; 12.0–12.5% – 21–22 days; 12.5–13.0% – 15–20 days and 13.0–13.4% – about 14.5 days

American Test at 65.5° also called 65.5°C Surveillance Test. This test was designed at the US Naval Powder Factory (now called the Naval Propellant Plant) by G.W. Patterson (Refs 2, 12 & 13) for testing the stability of smokeless propellants has been conducted at Picatinny Arsenal as follows:

For this test use a clean, dry 8-oz, colorless, special resistance glass, wide-mouthed bottle with velvet ground mushroom stopper which provides an air-tight seal. The bottle must be etched or ground on the side (1x2 inches) so as to be suitable for labeling. The alkalinity of glass must be not more than 0.02%, calcd as KOH. To determine the alkalinity, place 10g of ground glass in a Pyrex flask, add 20ml of N/20 HCl soln, allow to stand for 24 hrs and titrate with N/20 KOH, using phpht indicator

1) Transfer a 45g sample of propellant to the above bottle. If the proplnt is for a 10-inch or larger cannon, use a test sample of 5 proplnt grains, or as many as can be inserted in the bottle

Note: For small diam rocket proplnt sticks, cut a sufficient number of 3-inch lengths. For large diam sticks (up to and including 1¾ inches) cut a sufficient number from the length. For cylindrical sticks larger than 1¾ inches in diam, cut a longitudinal section thru the center, 1 inch wide on the grain surface, 1¾ inches in depth, and of sufficient length. For larger cruciform sticks, cut a longitudinal section from one arm, ¾ inch wide on the exterior grain surface and ¾ inch deep on the recession, and of sufficient length. Include lateral inhibitor, if present. For sheet proplnts, cut sufficient lengths 2½ inches wide, and coil

2) Stopper the bottle by simultaneously twisting and pressing on the stopper. Label the bottle so as to show the lot number of the proplnt and the date on which the test is begun

3) Place the bottle with contents in a chamber maintained at 65.5°±1°C (See Fig on p 28 of Ref 12 and on p 49 of Ref 13), the air in which is preferably maintained in constant circulation. After the bottle with contents has been heated for 24 hrs, reseat the stopper. Make daily observation of the appearance of the sample and note the number of days required to cause the liberation of visible reddish or brownish fumes of oxides of nitrogen

Note: It has been customary to consider any proplnt having a test value of 90 days or less to be of definitely impaired stability and any proplnt having a test value of 20 days or less to be sufficiently unstable as to require destruction or salvage

80° Surveillance Test is conducted in the same manner as the 65.5° test, except that the temp of the chamber is maintained at 80°±1°C. This test is made along with the 65.5° test in order to obtain more rapid (anticipatory) data

78°C Surveillance or Hot Storage Test. Accdg to Wiggam & Goodyear (Ref 9), the 65.5° test is too time consuming and they recommend conduct the test at 78°

75°C International Test. This test, which is a slightly modified procedure of Lenze & Pleus (Ref 4) can be used for HE's and pyro mixts. The following description is given in Ref 12, p 13 and in Ref 13, p 18:

Transfer a 10g portion of the sample as received to a weighing bottle 35mm in diam and 50mm high, cover with a watch glass, and weigh on an analytical balance. Heat the covered bottle with contents at 75° for 48 hrs, cool in a desiccator and weigh. Calc the loss in wt to percent and report as volatility of sample

Note whether the material has undergone decompn or is markedly volatile, as indicated by 1) Discoloration 2) Appearance of colored fumes above the sample 3) Development of an odor indicating acidity and 4) Loss of wt distinctly in excess of moisture content (as previously detd by a method appropriate to the material)

100° German Fume Test (Lagerung bei 100°). A 10g sample is weighed into a special glass-stoppered bottle and heated at 100°. The bottle is opened every 24 hours for 10 minutes to admit some air, and the heating continued until the appearance of brownish-red fumes

A powder is considered stable if it takes about 10 days to develop fumes; 3–5 days–not as good and below this, bad or very bad (Ref 6, pp 382–83)

75° German Fume Test (Warmlagermethode 75°). This test is suitable for Nitrocellulose and propellants:

1) Several 5g samples of expls are heated at 75° in glass-stoppered tubes, 28mm in diam and 200mm in length

2) The tubes are left open for 16 hrs and then heating is continued (with stoppers tightly closed) without interruption until distinct reddish-brown fumes appear

3) If the fumes do not appear in one week, the tube is opened for 10 minutes, to renew the supply of oxygen necessary to convert NO to NO_2, and the heating continued

This test was later improved by von Meerscheidt-Hüllessem (Ref 8) and was claimed to give very reliable results

The proplnt was considered very stable if it took not less than 130–150 days to develop reddish-brown fumes; stable – not less than 70–120 days; passable – not less than 50–60 days; unstable – at 10–40 days and very bad at less than 10 days. The latter proplnt had to be destroyed

Refs: 1) S. Thomas, ZAngewChem **2**, 1027 (1898) and **20**, 1143(1907) 2) G.W. Patterson, SS **5**, 47(1910) 3) 8th IntemlCongrApplied-Chem (1912), Appendix, Sectn **3**(b), p 311 4. F. Lenze & B. Pleus, SS **14**, 311–17(1919) 5) G. de Bruin, Rec **40**, 642(1921) and SS **17**, 59(1922) 6) F. Lenze & L. Metz, SS **23**, 382–83(1928) 7) A. Stettbacher, SS **24**, 172(1929) 8) F. von Meerscheidt-Hüllessem, SS **25**, 141(1930) 9) D.R. Wiggam & E.S. Goodyear, IEC(AnalEd) **4**, 73–6(1932) 10) Reilly(1938), 80–1 11)Clift & Fedoroff **2**(1943), p N22 (Thomas Test) 12) A.J. Clear, PATR **1401**(Rev 1), 13 & 28(1950) 13) A.J. Clear, PATR **3278**(1965), pp 22–3 & 49(65.5°C and 80°C Surveillance Tests) 14) Fordham (1966), 78–9 (Fume Test by firing a round in a part of a mine which can be completely cut off from the circulating air, followed by taking samples of resulting atm for analysis. Another method consists of firing the expl sample of known wt in a steel vessel reinforced with concrete, followed by removal of resulting gas for analysis. The common procedure to det N peroxide after a period sufficient to allow oxidation of N oxide. The Griess Ilosvay method is recommended; carbon monoxide can be estimated by reaction with iodine pentoxide)

Fumigène. Fr for a pyrotechnic smoke mixture. One of the pre-WWII French mixtures contd tetrachlorethane 40, ZnO 20, powdered Zn 15, Ca silicide 15 & Na chlorate 10%
Ref: Pepin Lehalleur(1935), 470

Fumigène, Compositions (Fr). The following compositions fumigènes (smoke compns) were patented in France in 1954 by Reure and published in 1956 by MP (See Ref)

Table

Ingredients %	Composition I	Composition Ib	Composition II	Composition III
Vinyl chloride	21.4	21	25	24.4
Diethylpentachloride	16.5	17	16	15.2
Diethylphthalate	3.6	–	2	–
K chlorate	34.8	36	27	–
Amm chlorate	–	–	–	27.5
Zinc	21.4	22	22	27.4
Trichlorobenzene	1.7	–	–	–
Dibutylphthalate	–	4	–	2.1
Hexachloroethane	–	–	–	3.6
Total	99.8	100	90	100.1

Ref: Germain Reure, FrP 1078608 (1954) & MP **38**, 419–21 (1956)

Fuming Nitric Acids. There are WFNA (white fuming nitric acid) and RFNA (red fuming nitric acid). They are described under Nitric Acid and in CondChemDict (1961), p 800-R; (1971), 618-R

Fumyl. A fine, buff-colored mixture used by the Russians during WWI as a smoke producing compn inside shrapnel shells. It consisted of TNT 45, AN 15 & Amm chloride 40%
Ref: M.M. Kostevitch, "Burning Ground", Imprim d'Art Voltaire, Paris (1927), p 36

Function on Arming Test, abbr as **FOA Test** is briefly described in Vol **4** of Encycl, p D1099-R and more fully by S.J. Odiemo, "Information Pertaining to Fuzes", Vol VII, PicArsn, Dover, NJ (1966), p III B-13

Function and Casualty Tests of Small Arms Ammunition. The purpose of these tests is to ascertain by firing in weapons of representative types whether the ammunition functions satisfactorily from the point of view of safety. Ammunition may be ballistically satisfactory, that is it may comply with individual performance specifications, such as velocity, pressure, accuracy, penetration, etc, yet be unfit for use in the field because of undesirable characteristics which jeopardize the safety of weapons

Detailed description of such tests is given in
Refs: 1) Anon, "Ordnance Proof Manual **7–15** 1945) 2) Anon, "Small Arms Ammunition", **TM 9–1305–200/TO 11A13–1–101** (1961), pp 17–21 (Surveillance)

Fungus Resistance Test for Fuzes is briefly described in Vol **4**, p D1094-L and fully described in US Standard MIL-STD-331 as Test No 110

Funkenchronograph. Ger for Spark Chronograph, such as Siemens invented in 1845. It is not described in this Encycl, Vol **3**, pp C304ff under CHRONOGRAPHS but is described in
Refs: 1) H. Kast, SS **8**, 90–1 (1913)
2) K. Cranz, "Lehrbuch der Ballistik", Springer, Berlin, Vol **3** (1929), p 27
3) H. Rumpf, Explosivstoffe **5**, 263 (1957)

Funkenkinematographie. Ger for Spark-cinematography

Funkenphotographie. Ger for Spark Photography

Funkenzünder. Ger for Spark Igniter, such as of Bornhardt, described in Beyling-Drekopf (1936), p 216

Functioning is the succession of normal actions from initiation of the first element to delivery of an impulse from the last element of the expl train (Ref 1). There are two types of functioning – high order and low order. The high order functioning consists of a deton, while low order functioning consists of a partial deton or a deflagration (burning). See Vol **4**, pp D245-R to D252-L. If high order deton is required, and only deflagration occurs, then there is a dud
Ref: 1) Anon, "Engineering Design Handbook, Ammunition Series; Fuzes", US Army Materiel Command Pamphlet **AMCP 706-210**(1969)

Functioning Tests of Detonators are described in US Army Specification **50-78-7**, p 8 and US Military Specification **MIL-D-2493**

Furacetic Acid. See Furylacrylic Acid, also known as Furfuralacetic Acid

Furaldehyde. See Furfural

Furan and Derivatives

Furan or Furfuran, OCH=CH–CH=CH; mw 68.075, colorless liq turning brown on standing; fr p –85.68°, bp 31.3°, fl p –40° (open cup), sp gr 0.937 at 20°, n_D 1.4214 at 20°, heat of combustion at const vol 500.1 cal; sl sol in w, sol in EtAc, methanol, alc, acet, eth, petrol eth, chlf, benz, toluene & ethylene glycol. According to Ref 2, first prepd before 1870 by distillation of 2-furoic acid from strong bases. Attenberg (Ref 3) obtained it from "Holzöl", a dry distillation product of pine trees. Ref 1 gives other methods. The laboratory method (Ref 4) involves heating *crude* 2-furoic acid. Industrially, furfural vapors and steam are passed over catalysts such as mixed Zn and Mn chromite at 400°. Furan dec at about 670°, or as low as 360° in the presence of Ni. It is very volatile and flammable. Furan is used in organic synthesis and may be catalytically hydrogenated to tetrahydrofuran. Inhalation of large quantities or lengthy skin contact is apt to produce toxic results (Ref 1, p [34] & Ref 7, p 1005). Unstabilized furan slowly forms an unstable peroxide on exposure to air, so precaution should be used when distilling it
Note: Accdg to US Rubber Co Report **No 5** (1948–49), p 15, furan reacted with Nitroform to give a small quantity of a solid product which burned vigorously and was extremely sensitive to shock. (The report is available in PicArsnLibrary as U21965)
Refs: 1) Beil **17**, 27, (16) & [34] 2) H. Limpricht, Ber **3**, 90(1870) & Ann **165**, 281 (1873) 3) A.Attenberg, Ber **13**, 879(1880) 4) H. Gilman & A.H. Blatt, OrgSyn, Vol **1**, 274(W.C. Wilson, Furan), J. Wiley, NY(1944) 5) O.W. Cass, ChemInds **60**, 612(1947) 6) DuPont Co New Products Bulletins(1947–1949), Wilmington, Del 7) A.P. Dunlop & F.N. Peters, The Furans, Reinhold, NY(1953), ACS Monograph No 119 8) CondChemDict (1961), 519-L 9) Kirk & Othmer, 2nd Ed, Vol **10**(1966), pp 247–48

1,2,3,4-Tetraiodofuran, OCI=CI–CI=CI; mw 571.68, mp 165°, expl 262°. Prepd by reacting aqueous I_2/KI with *1,2,3,4-tetra-HgCl-furan*

2-Nitrofuran, OCH=CH–CH=C(NO_2); mw 113.07, N 12.39%; yel-wh cryst(pet eth), mp 28°; sol in eth & w. Prepd by nitration of furan in Ac_2O
Refs: 1) Beil **17**, 28 2) R. Marquis, CR **132**, 141(1901)

3-Nitrofuran, OCH=CH–C(NO_2)=CH; mw 113.075, N 12.39%; crysts, mp 27°. Prepd by heating 3-nitro-2-furoic acid, which was prepd by oxidizing 3-nitro-2-methylfuran with $K_3Fe(CN)_6$/KAc at 100°
Refs: 1) Beil, not found 2) I.J. Rinkes, RecTravChim **57**, 390–4(1938) & CA **32**, 3980(1938)

5,5'-Dinitro-2,2'-difuran, $OC(NO_2)$=CH–CH=C–C=CH–CH=$C(NO_2)O$; mw 224.13, N 12.50%; yel-brn prisms, mp 213–4°. Found as a by product in the nitration of furan (Ref 2), and prepd intentionally by heating 5-nitro-2-bromofuran with Cu (Ref 3)
Refs: 1) Beil **19**, 32 2) R. Marquis, Ann-ChimPhys [8], **4**, 233(1905) 3) I.J. Rinkes, RecTravChim **50**, 981–8(1931) & CA **26**, 982(1932)

2,5-Dinitrofuran, $OC(NO_2)$=CH–CH=$C(NO_2)$; mw 158.075, N 17.72%, OB –40.5%; ndls(w) or prisms(alc), mp 101.5°; sol in eth. Prepd by nitration of 2-nitrofuran at 100° with 5g 1.2 nitric acid. It is an expl which is slightly less brisant and slightly more sensitive to impact than TNT. Heat of combustion is 432.2 kcal/m. Toxic to some people. Accdg to Gilman (Ref 4) it is an expl compd
Refs: 1) Beil **17**, 29 2) R. Marquis, CR **135**, 507(1902) 3) W. McPherson, USP 1827895(1932) & CA **26**, 849(1932) 4) H. Gilman, OSRD Rept **350**(1942)

Furan Peroxide, $C_8H_8O_4$; mw 168.15; crysts, dec 100°. Prepd from furan and oxygen at RT(Ref 2). The same author(Ref 3) later reported a different compd, a solid which expl on heating, resulted furan, O_2 & $CaCl_2$
Refs: 1) Beil, not found 2) G.O. Schenck, Naturw **31**, 387(1943) & CA **38**, 2336(1944) 3) G.O. Schenck, Ber **77B**, 661–8(1949) & CA **40**, 6075(1946)

Furan, Tetrahydro. See Tetrahydrofuran

1,2,3,4-Furan-Tetramercurous Acetate, $C_4O(HgAc)_4$; mw 1102.66, OB –36.3%; crysts, dec 226–8°. Prepd by treating furan with aq $HgAc_2$. By grinding with 50% KOH, filtering, washing with water, and drying in vacuo over sulfuric acid there is obtd a gray product which turns white and expl on heating to 210°; the gray material is more sensitive to heat than shock. By treating the tetra HgAc with hot, aqueous NaCl, there is obtd the tetra HgCl, dec 235°. If instead of furan, 2-furoic acid is reacted with $HgAc_2$, then CO_2 is liberated and a product contg some of the tetra HgAc, but analyzing C 14.03, H 1.99 & Hg 66.3%, ppts. When given the KOH treatment an expl gray compd results, analyzing C 5.64, H 0.64 & Hg 81.20% (which has less Hg than a tetra HgOH compd). On heating the gray material, an even more expl white material results, analyzing Hg 71.02%
Refs: 1) Beil, not found 2) R. Ciusa & G. Grilla, Gazz **57**, 323–9(1927) & CA **21**, 2686(1927)

Furanols and Derivatives

2-Furanol, OCH=CH–CH=COH; mw 84.07; crysts, mp 80°(dec 90°). Prepd by treating $OC(SO_3Na)$=CH–CH=$C\,CO_2Na$ with aqueous $NaOH/KClO_3$ in an autoclave at 200° for 15 hrs. The compd has a pungent, acrid odor, resinifies readily and turns brown on standing

3-Furanol, OCH=CH–C(OH)=CH; mw 84.07; crysts, mp 58°. Prepd either by Na/EtOH reduction of Na-Hg/base at 80° for 2 hrs reduction of 2-bromo-3-furanol (yel, mp 85°, prepd from bromination of 2-furoic acid). The method could not be reproduced (Ref 4)

5-Nitroso-2-furanol, OC(NO)=CH–CH=COH; mw 113.07, N 12.39%; yel crysts, mp 176°. Prepd by reacting 2-furanol with HNO_2 at 0°

2-Nitroso-3-Furanol, OCH=CH–C(OH)=CNO; mw 113.07, N 12.39%; brn-yel crysts, mp 151°. Prepd by reacting 3-furanol with HNO_2 at 0°

5-Nitro-2-furanol, $OC(NO_2)$=CH–CH=COH; mw 129.07, N 12.39%, OB –68.2%; yel crysts, mp 92°. Prepd by oxidizing the nitroso compd with $K_3Fe(CN)_6/KOH$ at 80°, or by direct nitration of 2-furanol with HNO_3/H_2SO_4 at 0°. It does not steam distil or reduce Tollens Reagent

2-Nitro-3-furanol, OCH=CH–C(OH)=CNO_2; mw 129.07, N 12.39%, OB –68.2%; yel crysts, mp

76°. Prepd by oxidation of the nitroso compd with $K_3Fe(CN)_6$/KOH at 80°. It steam distils but does not reduce Tollens Reagent

Refs: 1) Beil, not found 2) H.H. Hodgson & R.D. Davies, JCS **1939**, 806–9 & CA **33**, 6302(1939)(Parents) 3) Ibid, 1013–14 & CA **33**, 6833(1939) 4) P.S. Bailey & J.V. Waggoner, JOC **15**, 159–61(1950) & CA **44**, 4457(1950)

Furfural and Derivatives

2-Furfural, Furfuraldehyde, Furfurol Antoil or *2-Furaldehyde* (2'-Oxo-2-methyl-furan or 2-Formyl-furan in Ger), OCH=CH–CH=CCHO; mw 96.09; colorless liq when pure but red-brown on exposure to light or air; fr p –38.7°, bp 161–2°(760mm), 77°(37mm), 60–1°(18mm), fl p 68.3°(56.8°) – open cup, 140° – closed cup, sp gr 1.1544 at 25°, n_D 1.52345 at 25°, ignition temp 315–57°, lower expl limit at 125°, 2.1% v/v in air, heat of combustion at constant pressure and volume 559.8cal; sol in w, alc & eth. Apparently first prepd by Döbereiner (Ref 2) as a by product of prepn of formic acid from sugar, sulfuric acid and pyrolusite, who called it "Künstliches Ameisenöl" but did not determine a formula. Stenhouse (Ref 3) prepd an identical oil in 1840 by digesting wheat grains with sulfuric acid but without the MnO_2; he analyzed it and established the empirical formula as $C_5H_4O_2$. Ref 1 lists many subsequent prepns. A convenient laboratory prepn from corncobs is given in Ref 8. Industrial prepn started in 1922 by Quaker Oats at Cedar Rapids, Iowa. Modern industrial prepns start with agricultural residues such as oat hulls, corn cobs, rice hulls, bran, etc, which have a relatively high pentosan content (Ref 10). In these processes pentosans are hydrolyzed to pentoses by acid digestion, and these split out water internally to form furfurals:

$HOCH_2(CHOH)_3CHO \rightarrow$ OCH=CH–CH=CCHO + 3HOH.

Furfural is used for making other derivatives of 5-membered rings, as a solvent in refining of lubricating oils, and to form copolymer resins with phenol

Analytical procedures are found in Refs 6, 7, 12 & 14. Furfural may be detected by adding a few drops of aniline in acetic acid; an intense red color develops at once if present. Such other furfurals as the 5-methyl and 5-hydroxymethyl will also give a positive test. Furfural has low toxicity providing adequate ventilation and skin protection against contact is used

Refs: 1) Beil **17**, 272, (145) & [305]
2) J.W. Döbereiner, Ann **3**, 141(1832)
3) J. Stenhouse, Ann **35**, 301(1840)
4) The Miner Laboratories, Furfural and its Derivatives, Bull(1928), Chicago, Ill
5) C.D. Hurd et al, JACS **55**, 1082–84(1933) & CA **27**, 1880(1933) 6) E.E. Hughes & S.F. Acree, IEC(AnalEd) **6**, 123(1934) & CA **28**, 2646(1934) 7) A.P. Dunlop & F. Trimble, IEC(AnalEd) **11**, 602(1939) & CA **34**, 56(1940) 8) H. Gilman & A.H. Blatt, OrgSyn, Vol **1**, p 280(R. Adams & V. Voorhees: Furfural), J. Wiley, NY(1944)
9) Quaker Oats, Physical Data on Furfural, Bull **203**, Chicago(1947) 10) W.L. Faith, D.B. Keyes & R.L. Clark, Industrial Chemicals, pp 349–53, J. Wiley, NY(1950)
11) F.M. Turner, CondChemDict, p 306(1950)
12) AOAC, Official Methods of Analysis, 7th ed, Washington, DC(1950) 13) CondChemDict(1961), 519-R 14) Kirk & Othmer **10**, 2nd ed, pp 237–51(1966)

3-Furfural or *3-Furaldehyde*, OCH=CH–C(CHO)=CH; mw 96.09; liq, bp 144° (732mm), 71–3°(53mm), 66–8°(39mm), sp gr 1.111 at 20°, n_D 1.4945 at 20°. Prepd by the Rosenmund reduction of the corresponding acyl chloride (Refs 2 & 3)

Refs: 1) Beil, not found 2) H. Gilman & R.R. Burtner, JACS **55**, 2903(1933) & CA **27**, 3932(1933) 3) K. Hayes, JACS **71**, 2581–2 (1949) & CA **43**, 7015(1949)

Furfuralacetic Acid. See Furylacrylic Acid

5-Nitrofurfural-2, $OC(NO_2)$=CH–CH=CCHO; mw 141.08, N 9.93%; crysts, mp 35–6°, bp 132–40 (12mm). First prepd by Gilman (Ref

2) by nitration of furfurylidene diacetate and acid hydrolysis of the intermediate nitro-diacetate in overall 31% yield. A better yield was obtained by Witte (Ref 3) by nitration of furfural in the presence of PCl_3

Refs: 1) Beil, not found 2) H. Gilman & G.F. Wright, JACS **52**, 2550–4(1930) & CA **24**, 3508(1930) 3) M. Witte & C.J. Lind, USP 2502114(1950) & CA **44**, 5393(1950) 4) C.D. Nenitzescu & C. Bucur, RevChim, AcadRépPopulaireRoumaine **1**, No 1, 155–64 (1956) & CA **52**, 11811(1958)

5-Nitrofurfural-3, $OC(NO_2)=CH-C(CHO)=CH$, mw 141.08, N 9.93%; crysts, mp 76°. Prepd by nitration of furfurylidene-3-diacetate

Refs: 1) Beil, not found 2) H. Gilman & R.R. Burtner, JACS **55**, 2903(1933) & CA **27**, 3932(1933)

Furfural-N-nitriminoguanidine,
$OCH=CH-CH=CCH=NNHC(NH_2)=NNO_2$; mw 197.165, N 35.52%; yel ndls(alc), mp 220–4° (dec). Prepd in 99% yield by refluxing furfural and nitriminoguanidine in dilute alcoholic HAc at 100° for 15 mins

Refs: 1) Beil, not found 2) W.B. Stillman & A.B. Scott, USP 2416233(1947) & CA **41**, 3488(1947) 3) W.D. Kumler & P.P.T. Sah, JAmPharmAssoc **41**, 375–9(1952) & CA **47**, 2132(1953)

5-Nitro-furfural-N-nitriminoguanidine,
$OC(NO_2)=CH-CH=CCH=NNHC(NH_2)=NNO_2$; mw 242.15, N 34.71%, OB –66.1%; yel ndls (alc), mp 249–50°(dec). Prepd in 95% yield by refluxing 5-nitrofurfural with nitriminoguanidine in dil alc HAc at 100° for 15 mins

Refs: 1) Beil, not found 2) W.B. Stillman & A.B. Scott, USP 2416233(1947) & CA **41**, 3488(1947) 3) W.D. Kumler & P.P.T. Sah, JAmPharmAssoc **41**, 375–9(1952) & CA **47**, 2132(1953)

α-Furfuraloxime (Prior to 1924 thought to be the *β*-), $OCH=CH-CH=C-\underset{H}{\overset{OH}{C}}=N$; mw 111.10, ndls, mp 75–6°; sol in w, alc, eth & benz. Prepd from furfural, $H_2NOH.HCl$ & base in alc/w. Heating in soln converts it to the *β*-form

β-Furfuraloxime (Prior to 1924 thought to be the *α*-), $OCH=CH-CH=C-\underset{H}{\overset{OH}{C}}=N$; mw 111.10; ndls, mp 91–2°, bp 98°(9mm); sol in alc, eth, CS_2, benz & HAc. Prepd from furfural, $H_2NOH.HCl$ & base in alc/w

Refs: 1) Beil **17**, 281–2 & [311] 2) K. von Auwers & B. Ottens, Ber **57B**, 446–61 (1924) & CA **18**, 2509(1924) 3) O.L. Brady & R.F. Goldstein, JCS **1927**, 1961 & CA **21**, 3618(1927)

Furfuraloxime Peroxide,
$(OCH=CH-CH=CCH=NO\rightarrow)_2$: mw 220.19, N 12.72%; crysts, expl 130°. Prepd by hypochlorite oxidation of the oxime at 0°

Refs: 1) Beil **27**, 463 2) G. Ponzio & G. Busti, Gazz **36** II, 338–44(1906) & CA **1**, 842(1907)

5-Nitro-furfuraloxime,
$OC(NO_2)=CH-CH=CCH=NOH$; mw 156.10, N 17.94%; crysts, mp 154°(121°). Prepd by nitration at 0° of the oxime (Ref 4). For information concerning the isomeric nature see Ref 3

Refs: 1) Beil, not found 2) H. Gilman & G.F. Wright, JACS **52**, 2550–4(1930) & CA **24**, 3508(1930) 3) R.F. Raffauf, JACS **68**, 1765–6(1946) & CA **40**, 6992(1946) 4) C.D. Nenitzescu & C. Bucur, RevChim, AcadRép-PopulaireRoumaine **1**, No 1, 155–64(1956) & CA **52**, 11811(1958)

5-Nitro-furfural Semicarbazone,
$OC(NO_2)=CH-CH=CCH=NNHCONH_2$, mw 198.14, N 28.28%; crysts, mp 240°(dec). Prepd in nearly 100% yield by reacting the nitrofurfural with semicarbazide & HCl in dil alc NaAc (Ref 2)

Refs: 1) Beil, not found 2) W.B. Stillman & A.B. Scott, USP 2416234(1947) & CA **41**, 3489

(1947) 3) T. Takami & M. Ueno, JapP 180146 (1949) & CA **46**, 4029 (1952) 4) C.D. Nenitzescu & C. Bucur, RevChim AcadRépPopulaire Roumaine **1**, No 1, 155–64 (1956) & CA **52**, 11811 (1958)

5-Nitro-furfural-3-semicarbazone, mw 198.14, N 28.28%; yel ndls, mp 215°(dec, sl impure); sl sol in w. Prepd by heating 5-nitro-furfurylidene-3-diacetate with semicarbazide.HCl
Refs: 1) Beil, not found 2) H. Gilman & R.R. Burtner, JACS **55**, 2903 (1903) & CA **27**, 3932 (1933) 3) K. Hayes, JACS **71**, 2581–2 (1949) & CA **43**, 7015 (1949)

Furfuryl is the monovalent radical C_5H_5O– derived from Furfural. Exists in alpha- and beta- isomeric forms
Ref: Hackh's (1944), 361-R

Furfuryl Alcohol or *Furylcarbinol*, (2 -Oxy-2-methyl-furan in Ger), $\underline{OCH{=}CH{-}CH{=}C}CH_2OH$; mw 98.10; oily, nearly colorless when pure, amber in technical grade, bp 171°(750mm), 74.0–0.5° (17mm), fl p 75°(open cup), sp gr 1.12 at 25°, n_D 1.4845 at 20°, heat of combustion at const vol 6206.6cal/g. First prepd in 1860 by Ulrich (Ref 2) by treating furfural with alcoholic KOH. Beilstein (Ref 1) lists several methods. OrgSyn (Ref 6) uses the furfural-concd NaOH method. In 1934 Peters, Adkins & Connor (Ref 10) developed an industrial method of high pressure hydrogenation of furfural. Wojcik (Ref 7) describes various catalytic hydrogenation methods, mixed oxides of copper and chromium being found suitable. The alcohol is very sol in w when fresh, but becomes insol with aging or exposure to acid; otherwise sol in alc, eth, benz & chlf. Largest use is for dark thermosetting resins. Addition of 98–100% formic acid may explosively polymerize the alcohol (Ref 5a). Stronger organic and mineral acids will cause it to explode; the low vapor pressure (18mm at 40°C) and slight absorption thru the skin present no great toxicity hazard; ingestion causes the usual alcohol problems (Refs 11 & 13). It was used during WWII by the Germans under code name *SV–Stoff* to initiate the combstn of gasoline at the moment of its contact with nitric-sulfuric acids (Ref 6a) (See also under "Rocket Propellants, Liquid")
Refs: 1) Beil **17**, 112, (56) & [113] 2) Ulrich, Jahresber **1860**, 269 3) E. Erdmann, Ber **35**, 1855 (1902) 4) E.C. Hughes & J.R. Johnson, JACS **53**, 745 (1931) & CA **25**, 1245 (1931) (Spectrochemistry) 5) A.P. Dunlop & F. Trimble, IEC(AnalEd) **11**, 602–3 (1939) & CA **34**, 56 (1940) (Determination in presence of furfural) 5a) W.C. Tobie, NewsEd(AmChemSoc) **18**, 404 (1940) & CA **34**, 4571 (1940) 6) H. Gilman & A.H. Blatt, OrgSyn **1**, p 276 (W.C. Wilson: Prepn of 2-furylcarbinol), J. Wiley, NY (1944) 6a) CIOS Rept **30–115** (1946), p 11 7) B.H. Wojcik, IEC **40**, 210 (1948) & CA **42**, 3388 (1948) 8) F.M. Turner, CondensedChemical Dictionary, Reinhold, NY (1950), p 307 9) P. Aubertein, MP **32**, 411–416 (1950) 10) Kirk & Othmer **6** (1951), p 1002 11) S. Krop, JetPropn **24**, 224–5 (1954) & CA **48**, 13221 (1954) 12) J. Issoire, MP **42**, 333–42 (1960) & CA **55**, 14421 (1961) (Prepn from furfural & formaldehyde with NaOH in 80% yield – Cannizzaro reaction) 13) CondChemDict (1961), 520-R 14) Kirk & Othmer, 2nd Ed **10** (1966), 245–47

Furnaces and Ovens. *Furnace* is an apparatus or a chamber made of refractory material serving for heating, fusing or hardening materials by exposing them to high temps. The heat may be produced by burning gas, oil alcohol, coal, wood or by electricity. *Oven* is an apparatus or chamber heated to lower temps than furnace. Used in the laboratories and in the plants for drying various materials, including expls and proplnts
Refs: 1) Hackh's (1944), 361-R & 362 (Furnace); 603-R (Oven) 2) Perry (1950), 1596–1626 and 4th edition (1963), 9-34ff 3) Kirk & Othmer **7** (1951), 1–37 and 2nd Ed, Vol **10** (1966), pp 252–94 (Furnaces by M.H. Mawkinney)

2-Furoamide, OCH=CH–CH=CCONH$_2$; mw 111.10, N 12.61%; ndls (by subln), mp 142–3° (subl 100°). Prepd from the *acid chloride* and ammonia
Refs: 1) Beil **18**, 276 & [268] 2) L. Bodart, CR **43**, 393(1856)

3-Furoamide, OCH=CH–C(CONH$_2$)=CH, mw 111.10, N 12.61%; crysts, mp 169°. No prepn given, presumably by treatment of the ethyl ester or acid chloride with ammonia
Refs: 1) Beil, not found 2) H. Gilman & R.R. Burtner, JACS **55**, 2893–6(1933) & CA **27**, 3932(1933)

5-Nitro-2-furoamide, OC(NO$_2$)=CH–CH=CCONH$_2$; mw 156.10, N 17.95%; crysts (alc), mp 161.5–2.0°; sol in alc. Prepd from the acid chloride and ammonia
Refs: 1) Beil **18**, 288 2) R. Marquis, CR **137**, 520(1903) 3) C.D. Nenitzescu & C. Bucur, RevChim, AcadRépPopulaireRoumaine **1**, No 1, 155–64(1956) & CA **52**, 11811(1958)

5-Nitro-N-nitro-N-methyl-2-furoamide, OC(NO$_2$)=CH–CH=CCON(NO$_2$)CH$_3$; mw 215.12, N 19.54%, OB –63.3%; crysts, mp 87–90°. Prepd by nitration at 0° of the 5-nitro-N-methyl-2-furoamide
Refs: 1) Beil, not found 2) H. Gilman & H.L. Yale, JACS **72**, 3593–5(1950) & CA **45**, 1110 (1951)

Furodiazoles. See Oxdiazoles

Furoic Acid and Derivatives

2-Furoic Acid or *Pyromucic Acid* (Brenzschleimsäure in Ger), OCH=CH–CH=CCO$_2$H; mw 112.09, plates (w) or ndls (by subln), mp 133–4°(subln 100°), bp 230–2°; sol in w, alc & eth; heat of combustion at const vol 494.4kcal/m. First observed in 1780 by Scheele in the dry distillation of HO$_2$C(CHOH)$_4$CO$_2$H (*mucic acid*). Prepd from similar source material (Ref 1) until Erdmann (Ref 2) oxidized furfuryl alc with KMnO$_4$ or ammoniacal silver soln
Refs: 1) Beil **18**, 272 2) E. Erdmann, Ber **35**, 1858(1902)

3-Furoic Acid, OCH=CH–C(CO$_2$H)=CH; mw 112.09, plates (w), mp 121–2°(subl 110°); sol in alc, EtAc & hot w; steam distils. Found in the root of Phaseolus multiflorus (kidney bean) and the root rind of Evonymus atropurpureus
Ref: Beil **18**, (439)

3-Nitro-2-furoic Acid, OCH=CH–C(NO$_2$)=CCO$_2$H; mw 157.08, N 8.92%, OB –66.2%; crysts, mp 125°. Prepd by oxidizing 3-nitro-2-methylfuran with K$_3$Fe(CN)$_6$/KAc at 100°
Refs: 1) Beil, not found 2) I.J. Rinkes, RecTravChim **57**, 390–4(1938) & CA **32**, 4980(1938)

5-Nitro-2-furoic Acid, OC(NO$_2$)=CH–CH=CCO$_2$H; mw 157.08, N 8.92%, OB –66.2%; yel plates (w), mp 184–5°; sol in alc, eth & boiling w. Prepd by heating furan-2,5-dioic acid with nitrosylsulfuric acid at 100°(Ref 2); hydrolysis of 5-nitro-2-cyano-furan (Ref 5) and oxidation of 5-nitro-furfural with CrO$_3$ (Ref 4). Forms a **Silver salt** which expl on heating (Ref 3)
Refs: 1) Beil **18**, 287 & [269] 2) A. Klinkhardt, JPr [2] **25**, 51(1882) 3) B. Priebs, Ber **18**, 1363(1885) 4) H. Gilman & G.F. Wright, JACS **52**, 2550–4(1930) & CA **24**, 3508(1930) 5) C.D. Nenitzescu & C. Bucur, RevChim, AcadRépPopulaire-Roumaine **1**, No 1, 155–64(1956) & CA **52**, 11811(1958)

5-Nitro-3-furoic Acid, OC(NO$_2$)=CH–C(CO$_2$H)=CH; mw 157.08, N 8.92%, OB –66.2%; crysts, mp 138°. Prepd by nitration of 3-furoic acid or saponification the the ethylester (mp 56°) prepd by nitration of ethyl-3-furoate
Refs: 1) Beil, not found 2) H. Gilman & R.R. Burtner, JACS **55**, 2893–6(1933) & CA **27**, 3932(1933)

5-Nitro-nitrato ethyl-2-furoate, $OC(NO_2)=CH-CH=CCO_2CH_2CH_2ONO_2$; mw 246.13, N 11.38%, OB −58.5%; crysts, mp 81–2°(CH_3OH adduct), 124–5°(CH_3OH.HAc adduct). Prepd from the alcohol by nitration with HNO_3/Ac_2O at (−)10 – (−)5°. Obtd as the CH_3OH.HAc adduct, and may be freed of the HAc by treatment with pyridine
Refs: 1) Beil, not found 2) H. Gilman & H.L. Yale, JACS **72**, 3593–5(1950) & CA **45**, 1110(1951)

Furoyl is the monovalent radical CH:CH.O.CH:C.CO– of Furoic Acid (qv). Do not confuse with *Furfuryl* (qv) and *Furyl* (qv)
Ref: Hackh's(1944), 362-R

2-Furoyl Azide, $OCH=CH-CH=CCON_3$; mw 137.10, N 30.65%; plates(eth), mp 62.5°(dec 110°, expl 182–3°); sol in alc, eth, chlf, acet, HAc & benz; insol in w. Prepd from the hydrazide and $NaNO_2$/HAc (Ref 2). Used as a blowing agent in prepn of resinous foams contg glycidyl polyethers of dihydric phenols (Ref 4)
Refs: 1) Beil **18**, 281 2) P. Freundler, BullSoc [3] **17**, 423(1897) 3) H.L. Yale et al, JACS **75**, 1933–42(1953) & CA **48**, 4544 (1954) 4) H.L. Parry & B.O. Blackburn, USP 2739134(1956) & CA **50**, 5798(1956)

2-Furoyl Hydrazide, $OCH=CH-CH=CC(O)NHNH_2$; mw 126.11, N 22.22%; prisms(alc), mp 80°, bp 279°(dec); sol in w (over 50g/100ml), alc, chlf, benz & tol; dec in air. Prepd by refluxing ethyl furoate with hydrazine hydrate (Ref 2)
Refs: 1) Beil **18**, 279 2) P. Freundler, BullSoc [3] **17**, 423 (1897) 3) G. Carrara et al, Gazz **82**, 652–70(1952) & CA **48**, 6425 (1954)(Prepn)

5-Nitro-2-furoyl-hydrazide, $OC(NO_2)=CH-CH=CC(O)NHNH_2$; mw 171.11, N 24.56%; crysts, mp 170–1°(dec). Prepd from the acid and hydrazine hydrate in hot alc
Refs: 1) Beil, not found 2) H.L. Yale et al, JACS **75**, 1933–42(1953) & CA **48**, 4541(1954)

2-Furoyl Hydroxamic Acid (Brenzschleimsäure-hydroxylamid, in Ger), $OCH=CH-CH=CC(O)NHOH$; mw 127.10, N 11.02%; ndls(w), mp 128°; insol in eth; sol in w, alc & HAc. Prepd from ethyl furoate and H_2NOH in alc
Refs: 1) Beil **18**, 279 & [268] 2) R.H. Pickard & A. Neville, JCS **79**, 847(1901)

5-Nitro-2-furoyl hydroxamic acid, $OC(NO_2)=CH-CH=CC(O)NHOH$; mw 172.10, N 16.28%, OB −65.1%; crysts, mp 169°(dec). Prepd by treating the acid chloride with $H_2NOH.HCl$. Decomposes on treatment with nitric acid at 0°
Refs: 1) Beil, not found 2) H. Gilman & H.L Yale, JACS **72**, 3593–5(1950) & CA **45**, 1110(1951)

5-Nitro-2-(o-methyl)-furoyl hydroxamic acid, $OC(NO_2)=CH-CH=CC(O)NHOCH_3$; mw 186.125, N 15.05%; crysts, mp 151–2°. Prepd by treating the acid chloride with H_2NOCH_3. Decomposes on treatment with nitric acid at 0°
Refs: 1) Beil, not found 2) H. Gilman & H.L. Yale, JACS **72**, 3593–5(1950) & CA **45**, 1110(1951)

Furoyl Peracid or **2-Furoperacid,** $OCH=CH-CH=CCO_2OH$; mw 128.09; colorless ndls, mp 59.5°(dec)(may expl at 40°). Prepd by shaking an eth soln of difuroyl peroxide at −5° with acet-free methanol/$NaOCH_3$, and recryst from CCl_4 at −5°. Most stable at 0°, decompd by 360–450nm UV light and by inorganic or organic solids
Refs: 1) Beil, not found 2) N.A. Milas & A. McAlevy, JACS **56**, 1219–25(1934) & CA **28**, 4413(1934)

2,2′-Difuroyl Peroxide, $OCH=CH-CH=C-CO_2O_2C-C=CH-CH=CO$; mw 222.155; yel ndls(alc), mp 86–7°(dec); sol in eth, acet, HAc, chlf, methanol, CCl_4 & benz; insol in w & petr eth. Prepd from the

furoyl chloride and Na_2O_2 at 0°. Ordinarily stable but expl on heating over open flame; by impact or shock
Refs: 1) Beil **18**, [267] 2) H. Gelissen & J.D. Van Roon, RecTravChim **43**, 362(1924) & CA **18**, 2515(1924) 3) N.A. Milas & A. McAlevy, JACS **56**, 1219–25(1934) & CA **28**, 4413(1934)

2-Furoyl Nitrile, OCH=CH–CH=CCN; mw 93.09, N 15.05%; colorless oil (browns in air), bp 146–8°, 51°(23mm), sp gr 1.0790 at 25°, n_D 1.4739 at 25°; sol in alc & eth. Prepd by dehydrating the aldoxime with boiling Ac_2O
Refs: 1) Beil **18**, 278 & [268] 2) P. Douglas, Ber **25**, 1313(1892)

5-Nitro-2-furoyl Nitrile, $OC(NO_2)$=CH–CH=CCN; mw 138.09, N 20.29%; mp 65°. Prepd by dehydrating the aldoxime with boiling Ac_2O
Refs: 1) Beil, not found 2) C.D. Nenitzescu & C. Bucur, RevChim, AcadRépPopulaire-Roumaine **1**, No 1, 155–64(1956) & CA **52**, 11811(1958)

Furth Equation of State. See Vol **4** of Encycl, p D277

Furyl is the monovalent radical $-C_4H_3O$. Two isomers, alpha- and beta-, exist. Do not confuse with *Furfuryl (qv) and*
fuse with *Furfuryl* (qv) and *Furoyl* (qv)
Ref: Hackh's(1944), 262-R

Furylacrylic Acid and Derivatives
Furyl-β-acrylic Acid (Furfuralacetic Acid or Furacetic Acid), O.CH:CH.CH:C.CH:CH.CO_2H; mw 138.12. Exists as stable and labile isomers. The *stable* form: ndls(w), mp 141°, bp (with quick heating) 255–65°(286°); sol in alc, eth, benz, HAc & hot w; steam distils. Can be prepd by refluxing furfural with NaAc (Refs 1, 2 & 4). The *labile* form: prisms & plates (benz), mp 103–04°; sol in benz & hot w. Was prepd by refluxing furfuridine malonic acid with Ac_2O and recrystn as its piperidine salt from benz (the salt of the stable form is more sol in benz (Ref 3)

Furylacrylic acid can be used for prepn of some expls (See below)

Refs: 1) Beil **18**, 300–01, (440) & [273] 2) A. Bayer, Ber **10**, 357(1877) 3) C. Liebermann, Ber **27**, 286(1894) & **28**, 131(1895) 4) CondChemDict (1961), 521-R; (1971), 406-R

5-Nitro-β-furylacrylic Acid, O.$C(NO_2)$:CH.CH:C.CH:CH.CO_2H; mw 183.12, N 7.65%; no props are reported. Can be prepd by nitrating furyl-β-acrylic acid with nitric acid in Ac_2O

Its lead salt is expl
Refs: 1) Beil, not found 2) R. McGill, OSRD Rept **830** (1942), p 34

Lead 5-Nitro-furyl-β-acrylate, $(OC(NO_2)$:CH.CH:CCH:$CHCO_2)_2Pb$; mw 571.42, OB –64.4%; orn solid, mp(blackens at 170°). Prepd by treating with lead acetate the 5-Nitro-furyl-β-acrylic Acid (Ref 2). It is an expl comparable in sensitivity to TNT, but much less powerful (<50% TNT, by ballistic mortar test). Its thermal stability is satisfactory and it is only moderately hygroscopic
Refs: 1) Beil, not found 2) R. McGill, OSRD **830**(1942), p 34

Furyl-β-acrylic Acid Peroxide, $(O.CH:CH.CH:C.CH:CH.CO_2)_2$, mw 274.23, O 35.01%; wh ndls (turn yel on standing), mp 104°; sol in acet & EtAc; sl sol in eth. Prepd from furyl-β-acryloyl chloride and NaOH. Expl on heating
Refs: 1) Beil, not found 2) N.A. Milase & A. McAlevy, JACS **56**, 1219–25(1934) & CA **28**, 4413(1934)

Furyl Carbinol. See Furfuryl Alcohol in this Section

Furyl-β-(2,4,6-trinitro)-styrene, O.CH:CH.CH:C.CH:CH.$C_6H_2(NO_2)_3$, mw 305.21, N 13.77%; crysts, mp 128°. Prepd by condensing furfural with TNT in pyridine, using piperidine as catalyst
Refs: 1) Beil **17**, [67] 2) I.A. Pastak, Bull-SocChim **39**, 72–7(1926) & CA **20**, 3001(1926)

Fusain (Mother of Coal or Mineral Charcoal). See Vol 3 of Encycl, p C353-R and in Cond-ChemDict (1961), 521-R. It reduces the caking properties of the coal in which it occurs

Fuse. See FUSES

Fused Salts. Salts (ie ionic compds) in the molten state. High temps are usually involved in maintaining the molten condition. Common salt is the principal ingredient of many fused salts. They have been used as a base for circulating liquid fuels in nuclear reactors and for many other purposes
Refs: 1) CondChemDict (1961), 521-R & 522-L

Fusee. (Amer)(Pronounced "fu-zee"). An obsolete term for a fuse or detonator. At present, the name applies to a long-burning flare (about 20 minutes) used mostly for railroad emergency signalling. It has the appearance of a big candle and can be stuck into a wooden tie by means of a spike. It is ignited by scratching since a match type igniter forms a part of the fusee

An example of a military use is the Navy Light MK 1 Mod 1 (See Fig). This device is designed to produce a bright light for recognition and signalling purposes. The Mod number indicates the different colors of flame produced. For instance, Mod 1 gives off a blue signal for 75 seconds, whereas Mod 0 gives off a red signal and burns for 135 secs. The light can be seen up to 3 miles in clear weather
Refs: 1) GlossaryOrdn (1959), 125-R 2) Anon, "Pyrotechnic, Screening, and Marking Devices", **OP-2213**, Dept of Navy (1969), pp 5-29 & 5-51

Fusée. French word which corresponds to Amer fuze, fuse, fusee, flare or rocket
Ref: Same as Ref 1 under next item

Fusées. Following is the list of various Fr fusées:

Fusée antichar – Antitank rocket
Fusée d'artillerie – Artillery fuze
Fusée d'atterrissage – Landing flare
Fusée de Bickford – Bickford fuse
Fusée de bombe – Bomb fuze
Fusée à concussion – Concussion fuze
Fusée de culot – Base fuze
Fusée détonante – Detonating (or percussion) fuze
Fusée à double effet – Combination fuze
Fusée à durée ou Fusée à temps – Time fuze
Fusée à effet retardé – Delay fuze
Fusée éclairante – Illuminating flare
Fusée éclairante à parachute – Parachute flare
Fusée éclatante – Star flare
Fusée à étages – Multiple-stage rocket
Fusée fusante – Time fuze

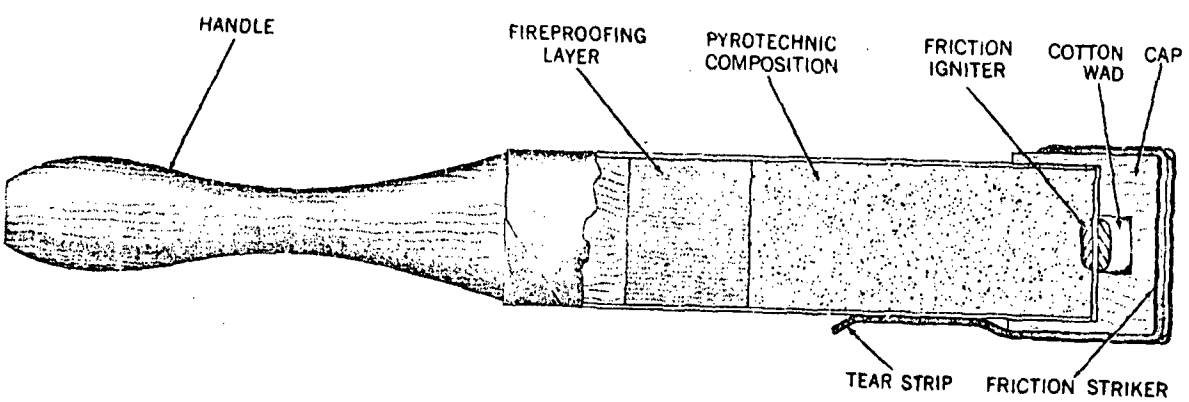


Fig Navy Light Mk 1 Mod 1

Fusée instantanée – Instantaneous fuze
Fusée lente – Slow match
Fusée d'ogive – Nose fuze (Point detonating fuze)
Fusée à obus – Shell fuze
Fusée percutante – Percussion (impact) fuze
Fusée à proximité radioélectrique – Proximity (or VT) fuze
Fusée retardée – Delay fuze
Fusée de signalisation – Signal flare or rocket
Refs: 1) A.M. Patterson, "A French-English Dictionary for Chemists", J. Wiley, NY (1921), 163 2) Anon, "Italian and French Explosive Ordnance", **OP1668**(1946), 185–89, US Bureau of Ordnance (Navy Dept)

Fusee-Type Flares. See Ohart (1946), p 306 and in this Vol, p F64, under Flares

Fusehead or Electric Matchhead. A flashing (igniting) element used in electric blasting caps and detonators. It is a combination of bridge wire, igniter bead (drop or cylindrical chge) and lead-in wires. There are several types, and the following are described in Vol 4 of Encycl:
p D138-R, Fig 8 – Low-tension Fusehead for Electric Detonator
p D739-R, Fig 10 – British Fusehead in Electric Delay Detonator
p D740-L, Fig 11 – British Fusehead in Electric Short Delay Detonator
p D741, Fig 12 – The sequence of events in firing Electric Detonator
p D807, F g 71 – Special Military Electric Cap M6 with wire bridge surrounded by cylindrical ignition chge of LSt/Ba chromate, followed by LA and RDX
p D808, Fig 72 – Medium Energy EBW (Exploding Bridge Wire) Detonator with Wollaston Wire
p D808, Fig 73 – EBW Plug (HV-1) Modified for Very High Voltage
p D809, Fig 74 – EBF Detonator Design for High Voltage Application
p D846, Fig 1-27 – Navy Electric Detonator, Mk46 Mod 0 with bridge wire located in a loose cylindrical charge contg 75/25–DAzDNP/K chlorate mix
p D847, Fig 1-28 – Navy Electric Detonator Mk51Mod0 with bridge wire located in flash chge XC-9
p D847, Fig 1-29 – Army Electric Detonator M36 with bridge-wire located in a cylindrical chge of MF milled in 2.4% soln of NS in butyl acetate
p D848, Fig 1-30 – Army Electric Detonator M48 with colloidal graphite bridge, which ignites Milled LA and this detonates Dextrinated LA and PETN
p D848, Fig 1-31 – Army Electric Detonator T20E1 with tungsten wire bridge, which ignites colloidal LA, and this detonates dextrinated LA & PETN
p D849, Fig 1-31 – Electric Delay Detonator T65 with colloidal graphite bridge, which ignites Lead Mononitroresorcinate, which is followed by Normal Lead Styphnate, 70/30-Lead Peroxide/Boron, 90/10-Barium Chromate/Boron, Dextrinated LA and RDX
p D850, Fig 1-33 – Button Type Electric Detonator T62 with colloidal graphite bridge, surrounded by Colloidal LA, which is followed by Dextrinated LA and PETN
p D855, Fig 1-43 – Navy Electric Fuze Primer Mk112Mod0 with bridge wire surrounded by flash charge XC-9, which is followed by base chge of 75/25-DAzDNPh/K chlorate
p D855, Fig 1-44 – Navy Electric Fuze Primer Mk121 with conductive carbon bridge, surrounded by ignition chge of LSt in lacquer followed by flash charge of XC-9 and base chge same as in Mk112Mod0
p D856, Fig 1-45 – Navy Experimental Spray Metal Electric Fuze Primer with bridge wire attached by "spray" metal (method developed at NOL and described on p D856-R), followed by ignition chge of dry LSt pressed at 3400psi, spacer and base chge of PETN

Accdg to Fordham (1966), p 116, the first successful fusehead was invented in Germany by Krannichfeldt. The "sandwich" type construction is now used in many countries, including GtBritain. It is illustrated in Fig 10.2 on p 117. The manuf of such fuseheads in GtBritain is described on pp 117-1 of Fordham. Assembly of electric detonators using these fuseheads is described on pp 119–21 with Fig 10.5

German fuseheads and their manuf are de-

scribed in PATR **2510**(1958), pp Ger 53 & Ger 54

Fusehead Bead of Ingram contained, in addition to a binding agent, an aromatic compd contg a nuclear-bound iodine atom attached to at least one terminal oxygen atom, such as iodoxy acid iodoso compds of benzene (toluene azido benzene or its derivatives). The expln point of these derivatives as determined by the 5 sec expln temp test (Ref 2) has not exceeded 300°F.

May be used for both low-tension and high-tension electric blasting initiators (Ref 1)

Refs: 1) L.K. Ingram, USP 2241496(1941) 2) A.J. Clear, "Standard Laboratory Procedures for Determining Sensitivity, Brisance and Stability of Explosives", PATR **3278** (1965), pp 7–8 & 39 with Fig 8

Fuseheads, Electrical Characteristics of A study was undertaken with a view to determine the basic mechanism of failure in series firing ability. The excitation time, transfer time and total time of three different types of squibs: (i) with LMNR as base (ii) with Lead Dinitro-ortho-cresol (LDNOC) as base and (iii) with charcoal plus potassium chlorate as base were determined

It was found that for the charcoal based squib, the transfer time was almost double the initiation time. This large transfer time obviously takes care of the variations in the excitation timings due to different factors in the manufacture of squibs. This ensures continuous flow of current thru all the squibs connected in series until the time the most sensitive squib breaks the circuit. In the case of LMNR and LDNOC fuseheads the transfer time was found to be quite low and the total time of some of the fuseheads was less than or equal to the excitation times of some others in the same group. Such squibs when connected in series caused failures in series firing. As a confirmation to this theory, a retardant was introduced in the LMNR fuseheads to increase the transfer time, and no misfires were noted

Ref: 1) K. Bhujanga Rao et al, R&D Dept of Indian Detonators Ltd, Hyderabad, India 2) Expls&Pyrots **4**(4), 1971

Fuseheads, Pressures Produced by Their Combustion was discussed by I. Ito et al of Univ of Kyoto, Japan in Suiyokaishi **15**(1), 29–32(1963) and abstracted in CA **62**, 12966(1965). The pressure produced by fusehead contg DAzDNPh was 70kg/sq cm, while the fusehead with mixt of PbDNResorcinate, Pb rhodanate & K chlorate produced 50kg/sq cm

Fuse, Hot Wire, Lighter Used in Pyrotechnics. See Vol **4**, p D762-L

Fuse Igniters or Lighters Used in Ordnance Items. See Vol **4**, pp D768 & D769-L

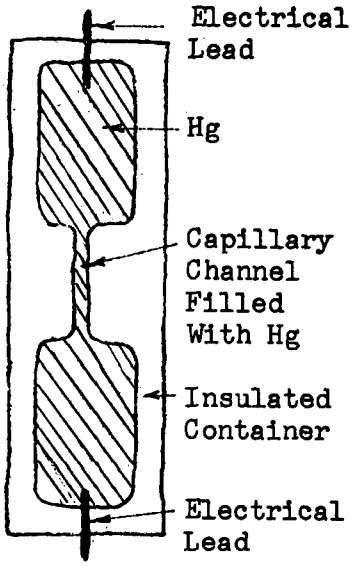
Fuse Lighter "Pyrocore". See Vol **4** of Encycl, p D763-L

Fuse Lighter "Pyrofuse". See Vol **4**, p D762-R

Fuse Lighters Used in Pyrotechnics. See Vol **4**, p D762

Fuse Lighter "Thermalite Ignitacord". See Vol **4**, p D762-R

Fuse, Liquid-Metal, Self-Healing. When mercury replaces the filament of an exploding-wire fuse, it will break contact by vaporizing upon application of an overvoltage, then make contact by condensing when the overload disappears. This happens within a heavy-walled capillary tube connecting two reservoirs (see sketch). Evaporation forces mercury from the capillary to break the circuit Experimental quartz glass and pyrex tubes exhibit da-



mage from heat and shock; sapphire and alumina do not. Typical response: 13 milliseconds delay in breaking contact, 1 millisecond to reestablish conduction. Geometry determines resistance: test models range from 0.1ohm to 20ohms. Investigator cites specific problems for future study
Refs: 1)C.A. Renton & R.J. Manco, "An Exploding Wire Self-Healing Fuse", pp 299–304 in: Conference on the Exploding Wire Phenomenon, W.G. Chace, Ed, Plenum Press, New York (1968) 2) Expls&Pyrots 1(11), 1968

Fusel Oil. A clear, colorless, volatile, poisonous, oily mixture with a disagreeable odor, consisting largely of amyl alcohols (See Vol 1 of Encycl, pp A394-R to A395-R), of which isoamyl (isobutyl carbinol) and active amyl alcohol (2-methyl-1-butanol) are chief constituents. Other alcohols, as well as aldehydes, esters and acids are present but no normal primary amyl alcohol (1-pentanol). Typical spec for refined product: sp gr 0.811–0.815 (20/20°), fl p (open cup) 123°F; distn range ca 115–135°; sol in w, alc & eth; miscible with gasoline. Can be obtd as a by-product in the alcoholic fermentation of starch- or sugar-contg materials, such as potatoes, beetroots, grapes, grain, etc. Used as gelatinizing agent for expls and proplnts and for many other purposes as listed in Ref
Ref: CondChemDict (1961), 522-L; (1971), 406-L

Fuse Match Lighter Used In Pyrotechnics. See Vol 4 of Encycl, p D762-L

FUSES

(Not to be confused with FUZES)

Fuses may be defined as tube- or cord-like containers filled with deflagrating or detonating materials. Both the outer sheathing material and the weight and type of expls contd will depend upon the required usage

Deflagrating expls or propellants burn whereas detonating expls detonate to produce blast, shock, and fragments. A fuse designed using deflagrating expls is known as a **safety fuse**, and that designed using detonating expls is known as **detonating fuse.** Care should be exercised in the use of fuses because ofthe difference in output of the two major types. Other characteristics which differentiate between deflagrating and detonating expls are: 1) reaction rate (usually a ±10% variation) 2) potential hazard 3) Department of Transportation shipping class 4) Department of Defense Compatibility Class 5) Department of Defense Hazard Class. The reaction rate of deflagrating expls (ie safety fuse) is normally 90 or 120 seconds per yard (Ref 2). Burning rate of delay cords depends upon composition. The potential hazard of this type is based on the fact that the rate of flame propagation increases with increasing pressure from gases released in a confined space. The shipping class is C, compatibility B, E, or H and hazard class 1 (Ref 1). With the detonating types, the reaction rate is normally 17000 to 26000 feet per second. Detonation rate of a particular expl increases with increased core density. The hazard inherent in this type is the high velocity fragments resulting from sheath rupture. Shipping class is A or C, compatibility class I or D and the hazard class 7 or 2(Ref 1). The major types of expls used in fuses are illus-

trated in Fig 1, A thru E
Refs: 1) Anon, "Safety Manual", **AMCR 385-100** (April 1970) 2) Sidney A. Moses, "Linear Explosives", Ordnance, Vol **LV1** No 311, 355-57 (March-April 1972)

Fuses, Bickford. See Vol **2** of Encycl, p B112-L

Fuses, Bickford Cordeau. See Vol **2** of Encycl, p B112-R

Fuses, Detonating. Detonating fuse, Type B material (Fig 1), is usually referred to by the trade name, *Primacord* (registered trade name of Ensign Bickford Co) (See Vol **2** of Encycl, p B112-R and Vol **3**, p C529-R). It is widely used for initiating expls in quarrying, mining and general blasting operations

Military uses include demolition-charge harnesses, warhead assemblies, and aerospace applications such as missile-stage separation and destruct systems. Approx 500 million feet per year of detonating fuse is manufd in the US, with worldwide consumption approaching 1 billion feet (Ref 2)

The expl core, contg either PETN or RDX is covered by various combinations of materials such as textiles, waterproofing compds, plastics, rubber, etc. The covering may be wire reinforced (Ref 2)

Type C materials (Fig 1) are smaller in diam and contain less expl. Four types are available: *Primalene* (Ensign Bickford Co), Mild detonating fuse (MDF), *Pyrocore* (du Pont Company) and delay fuse

Type D material (Fig 1) is made with a metal tube covered by a polyethylene sheath and many layers of woven fiberglas. It is called "controlled detonating fuse" (CDF) (Ref 2) and used mainly in aerospace applications to transfer a detonation stimulus from an initiating element to some remote expl function

Type E material (Fig 1) is called flexible linear shaped charge (FLSC). See shaped charge
Refs: 1) FM-25, "Explosives and Demolitions", Department of Army (October 1963) 2) Sidney A. Moses, "Linear Explosives", Ordnance Vol **LV1**, No 311, 355-57 (March-April 1972)

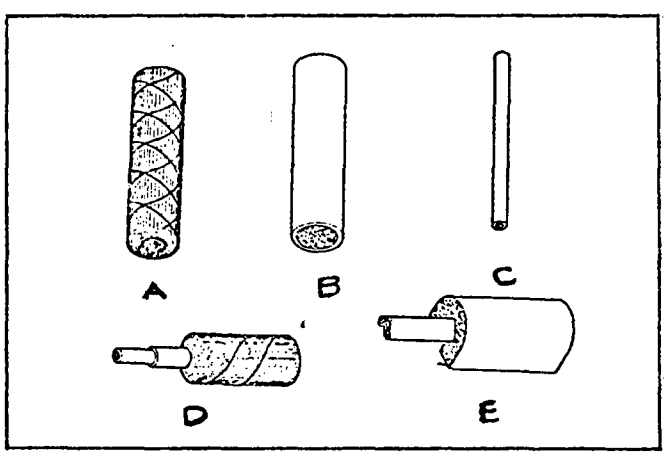


FIG 1

Fuses, Miner's. Same as Bickford Fuse described in Vol **2**, p B112

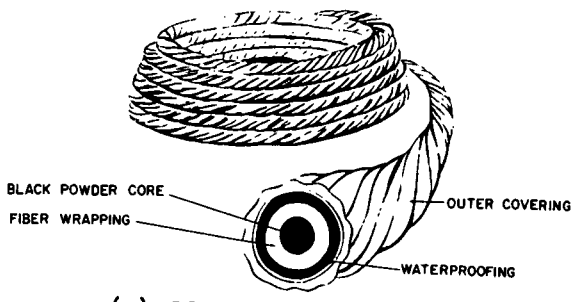
Fuses, Safety. The safety fuse (Type A, Fig 1), consists of a core of granular Black Powder sheathed with various protective layers. It is used mainly for mining and quarrying operations. The flame developed is transmitted along a preselected path to the pyrotechnic charge, where it can either initiate the chge directly or initiate one or several squibs that are used as chge igniters

Two types of fuse are in common use by the US Army: (1) blasting time fuse that has a spiral wrapped outer cover usually colored orange and (2) safety fuse M700 that has a smooth green plastic cover with length markers of abrasive material (so they can be felt in the dark). These two types of fuse are shown in Fig 2 (Ref 1)

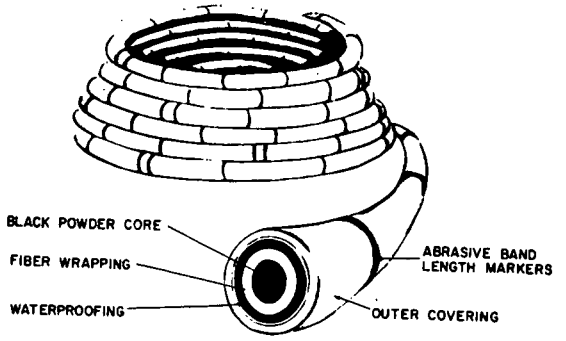
Safety fuse can be ignited in several ways:

1) Matches. Ordinary matches are frequently used to light a single line of fuse. The fuse must be split, taking care not to dislodge the BkPdr filler. The match is applied so that its initial flare ignites the fuse

2) Military Fuse Lighters. The military lighters provide a method of positive ignition of safety fuse even under extreme environmental conditions (See Vol **4** of Encycl, Sect



(a) Blasting Time Fuse



(b) Safety Fuse, M700

FIG 2

3, Part C, p D768-L)

Safety fuse should be used and stored so as to prevent contact of the covering from petroleum distillates that will attack the covering and make the fuse unreliable. Twists, kinks, or sharp bends may crack the covering or cause discontinuities in the powder train. Cold weather causes the covering to be less resistant to mechanical movement and additional care is required to maintain its reliability. When safety fuse is used in applications where it is closely confined, its burning rate is considerably increased, the tighter the confinement, the faster the burning rate

Ref: 1) FM-25, "Explosives and Demolitions", Department of Army (October 1963)

Fuses Described in the Book of Fordham include:

Detonating Fuse: Introduction (p 131); manuf by the dry process (pp 132–33 with Fig 12.1); manuf by the wet process (p 133); properties (p 134); LEDC (Low Energy Detonating Cord) (pp 134–35)

Safety Fuse: Introduction (p 136); manuf (pp 136–38 with Fig 13.1); properties (p 139); testings (pp 139–40)

Instantaneous Fuse (p 140)

For some purposes, especially in fireworks, when instantaneous, or almost instantaneous, transmission of flame is required without detonation, such a fuse is used. It consists of a highly combustible thread (often made from nitrated paper) inside a tube of known diam. The thread (serving as a core) occupies only a small part of the cross section of the tube, so that the gases can penetrate along it freely. The high speed of burning is caused by the rapid passage of hot gases along the tube, igniting the core well in front of the portion already consumed. The rate of burning is not easily controlled, but the uses to which the fuse is put are such that this is not of great importance. Threads coated with BkPdr may be used instead of nitrated paper

These fuses are usually made in small quantities by simple hand method

Ref: Fordham (1966), 131–40

Fuse Test. See Test a) in Vol 1, pp XXII–XXIII, under SENSITIVITY TO FLAME, HEAT, SPARKS, ELECTROSTATIC DISCHARGES, ETC

Fusible Alloy is an alloy melting at a temp lower than the mean mp of the constituents (Ref 1). The term is generally applied to alloys melting below 450°F(233°C). They can be binary, ternary, quaternary, or quinary mixts of Bi, Pb, Sn, Cd, Indium and less frequently other metals. Eutectic alloys are relatively few in number and are the particular compns that have definite and minimum mp as compared with other mixts of the same metals. Table I of Ref 2 lists 17 eutectics of mp's ranging from 46.89° to 248.0°C. Of these the Lipowitz's eutectic, melting at 70.0° is well known. It consists of Bi 50, Pb 27, Sn 13 & Cd 10%. Table II of Ref 2 lists 13 non-eutectic alloys with mp's ranging from 64.0 to 159°C. Table III of Ref 2 lists eight common fusible alloys of mp's 70 to 138°. Of

these *Wood's alloy* (mp 71°, contg Bi 50, Pb 24, Sn 14 & Cd 12%) and *Rose's alloy* (mp 100°, contg Bi 50, Pb 28 & Sn 22%) are well known in chem labs

The chief industrial uses of fusible alloys are for fusible automatic sprinklers, anchoring bearings and bushings, for heat transfer liquids, for glass to metal seals, etc (Ref 2)
Refs: 1) Hackh's Dict (1944), 363-L
2) CondChemDict (1961), 522–23; (1971), 30 (Alloy, fusible), 539 (Wood's metal)

Fusible Metals. Any metal or alloy of relatively low mp, such as Na, K, Pb, Sn, etc
Ref: Hackh's Dict (1944), 363-R

Fusil. Fr for rifle

Fusinit(e). A Ger coal (Kohle) illustrated in Ref
Ref: Ullmann **9** (1957), 168

Fusion. Usually a synonym for melting or flowing together, eg, the transformation of a solid into a liquid by the application of heat and w/o using a solvent other than a flux (Ref 1). Since melted substances tend to mix readily, the word has assumed the meaning of "melt" and "blend". The so-called *fusion* of protons to form helium as utilized in the *hydrogen bomb* indicates a union of two or more protons to form an element with great liberation of energy

A brief description of fusion- or hydrogen bomb (H-Bomb) is given in Vol **1** of Encycl, p A499, under Atomic (or Nuclear) Bomb
Refs: 1) Hackh's Dict (1944), 363-R
2) CondChemDict (1961), 523-R (Fusion); 815-L (Nuclear energy); 815-R (Nuclear fusion); (1971), 407 (Fusion)

Fusion Mixture. A mixt of Na and K carbonates used to fuse insol substances of high mp's (such as ores) in order to render them soluble in the form of double carbonates. Fusion can also be achieved by heating insol substances with Na or K hydroxides
Ref: Hackh's Dict (1944), 363-R

Fusion Reaction is described briefly in Vol **1** of Encycl, pp A500 to A504-L, under "Atomic (orNuclear) Energy; Atomic (or Nuclear) Explosions". This was described by C.G. Dunkle, formerly of Picatinny Arsenal

See also "Nuclear Reaction" in CondChemDict (1961), 815-R & 816-L (Compare with Fission Reaction)

Fusion Reactor. See Nuclear Reaction in CondChemDict (1961), 816-L; (1971), 632-R

Fuze Catalog of 1970 is an up-date of the 1959, Army, Navy, Air Force Fuze Catalog, which lists technical and reference info for all fuzes and fuze expl components, both standard and developmental. There are 2100pp divided into *Volume 1.* Current Fuzes (classified); *Volume 2.* Obsolete and Terminated Fuzes (unclassified); and *Volume 3.* Fuze Explosive Components (classified)
Refs: 1) MIL-HDBK-137, Fuze Catalog, 20 Feb, 1970 2) Expls&Pyrots **3** (8) (1970)

Fuze Detonator. Definition is given in Vol **4**, pp D836-R & D837-L and description of typical devices are on pp D840, D850 and Figs 1-22, 1-23, 1-24, 1-25, 1g26, 1-27, 1-28, 1-29, 1-30, 1-31, 1-32 & 1-33

Fuze, Flueric. See Fluidics for Ordnance, Vol **6**, p F112

Fuze, German (Zünder). Many fuzes were developed in Germany before and during WWII. They were subdivided into Bombenzünder (Bomb Fuzes) and Granatenzünder (Projectile Fuzes). They are listed, including numerous illustrations on pp Ger 54-R to Ger 63-R of PATR **2510** (1958). Five other refs are given on p Ger 63-R

Fuze Handbook, Up-dated The engineering textbook on fuze design is arranged in three parts: (1) Fundamental principles (covers purposes, general functioning, design considerations, initiation, and explosive train) (2) Basic arming actions (describes all arming principles) and (3) Fuze design (discusses the various types of fuzes, such as artillery, bomb, and mine and covers fuze testing)

The new edition updates the one written a dozen years ago. Among the new material: dual arming, design for rain insensitivity, analog computer analysis, electric power sources including RC circuits, electric, fluidic, and pneumatic arming, detached lever escapements, spiral unwinder, enlarged design guidance, and complete list of JANAF Journal Articles

Refs: 1) Glossary, Index, 214 pages, Engineering Design Handbook, "Fuzes", Army Materiel Command, **AMCP 706-210**, November 1969, AD-889245L, obtainable from Defense Documentation Center, Cameron Station, Alexandria, Va 22314 2) Expls&Pyrots **3**(6), 1970 3) Ibid, **5**(9), 172

Fuze Primers. See Vol **4**, pp D850 to D856 and Figs 1-34, 1-35, 1-38, 1-39, 1-40, 1-41, 1-42, 1-43, 1-44 and 1/45

FUZES

(Not to be confused with FUSES)

The definition of the term fuze is given in Vol **4**, p D879-L under Introduction; and definitions of various types of fuzes are given under the "List of Fuzes", pp D879-L to D884-R

The definition given by Ordn Dept after Spanish-American War was: "A mechanical device, with or without explosive elements, used to explode a shell, bomb or other type of projectile". The British name for an unloaded fuze is *pistol*

Military Fuzes may be subdivided into Artillery Fuzes, Demolition Items Fuzes, Hand Grenades Fuzes, Land Mines Fuzes and Bomb Fuzes

A. Artillery Fuzes (pp D885 to D887-L) may be subdivided into: PD(Point Detonating), PI(Point Initiating), T(Time), SQ(Superquick), TSQ(Time, Superquick), MT(Mechanical Time) and MTSQ(Mechanical Time, Superquick)

Following Figs are for various fuzes:
Fig 1-73 on p D887 is for PD, SQ Mk 27 Fuze
Fig 1-74 on p D889 is for PD, SQ, Delay, M5A1 Fuze
Fig 1-75 on p D890 is for *PD M52A1 Fuze*
Fig 1-76 on p D892 is for *PD, TSQ, M54 Fuze*
Fig 1-77 on p D893 is for *PD, M56 Fuze*
Fig 1-78 on p D894 is for *Time(Fixed)M65A1 Fuze*
Fig 1-79 on p D895 is for *PD, M75 Fuze*
Fig 1-80 on p D896 is for *TSQ, M77 Fuze*
Fig 1-81 on p D897 is for *PDCP, M78 Fuze*, where CP means Concrete-Piercing
Fig 1-82 on p D898 is for *Time, M84 Fuze*
Fig 1-83 on p D899 is for *PD, M89 Fuze*
Fig 1-84 on p D900 is for *PI, M90A1 Fuze*
Fig 1-85 on p D901 is for *PD, M503A1 Fuze*
Fig 1-86 on p D903 is for *PD, M557 Fuze*
Fig 1-87 on p D905 is for *PD, XM593 Fuze* in unarmed position
Fig 1-88 on p D906 is for the above Fuze in armed position
Fig 1-89 on p D907 ia doe *MT, M43A4 Fuze*
Fig 1-90 on p D911 is for *MTSQ, M548 Fuze*
Fig 1-91 on p D912 shows "explosive train" for the above fuze
Fig 1-91a on p D912 is for *MTSQ, M564 Fuze*

BD(Base-Detonating) Artillery Fuzes:
Fig 1-92 on p D913 is for *BD, M58 Fuze*
Fig 1-93 on p D914 is for *BD,M66A2 Fuze*
Fig 1-94 on p D914 is for *BD, M72 Fuze*
Fig 1-95 on p D915 is for *BD, M91A1 Fuze*
Fig 1-96 on p D916 is for *PIBD, M530A1 Fuze*
Fig 1-97 on p D918 is for *BD, M578 Fuze*

Proximity or VT(Variable Time) Artillery Fuzes:
Fig 1-98 on p D919 is for *Typical VT Fuze* and are listed on p D921 without Figs the following:
Fuzes, Proximity:
M504A1, M504A2, M513, M513A1, M513B1, M514, M514A1, M515, M516, M516A1, M516B1, M516B2, M517 and M532 for Mortars

Recoilless Rifles Projectile Fuzes.
They are similar to those used in ordinary artillery fuzes. Nineteen fuzes are listed on pp D921-R & D922-L, without giving their Figs

Mortar Projectile Fuzes.
They are similar to those used in artillery projectiles

Eighteen fuzes are listed on p D922 without giving the Figs

Foreign Artillery Fuzes of WWII.
British. Info is confidential
French. No info available
German. The name is *Geschusszünder* instead of erroneously varityped Gessoss-zünder

A complete list of German fuzes used during WWII, including their Figs, is given in:
1) TM 9–1985–3(1953), p 547–608 and
2) PATR **2510**(1958), p Ger 58 to Ger 64
(See also at the end of this Section)
Italian (Spoletti). The list of fuzes given on p D923 of Vol 4 includes those described in TM 9–1985–6(1953), pp 133–154
Japanese. The list of fuzes given on pp D924 & D925 includes those described in TM 9–1985–5(1953), pp 391–426 & 518–43
Russian (Vzryvateli and Snariadnyiye Trubki). More complete list than on p D925 is given in PATR **2145**(1955), pp Rus 7 & Rus 8
Spanish. No info
Swedish. No info
Swiss. No info

B. Fuzes for Bombs are briefly described in Vol 4, Section 6, Part C, pp D967 to D1010

They may be subdivided into:
Bomb Nose Fuzes, such as shown in:
Fig 4-1 on p D968 for *Fuze, AN-M103A1*
Fig 4-2 on p D970 for *Fuze, AN-Mk219*
Fig 4-3 on p D971 for *Fuze, Mk243Mod0*
Fig 4-4 on p D972 for *Fuze, M904E2*
Fig 4-5 on p D974 for *Fuze M197*
Fig 4-6 on p D975 for *Fuze, AN-M159*
Fig 4-7 & Fig 4-8 on p D977 for *Fuze, M157*
Fig 4-9 on p D978 for *Fuze, AN-M173A1*
Bomb Tail Fuzes, such as shown in:
Fig 4-10 on p D980 for *Fuze, AN-M100A2*
Fig 4-11 on p D982 for *Fuze, M115*
Fig 4-12 on p D983 for *Fuze, AN-Mk228*
Fig 4-13 on p D985 for *Fuze, M123A1*
Fig 4-14 on p D986 for *Fuze, M123A1* (Operation)
Fig 4-15 on p D987 for *Fuze, M132*
Fig 4-16 on p D989 for *Fuze, M906*
Bomb MT (Mechanical Time) Fuzes, such as shown in:
Fig 4-17a on p D991 for *Nose MT Fuze, AN-M146A1* (Unarmed) and
Fig 4-17b on p D992 for above fuze (Armed)
Fig 4-18 on p D994 for *Nose MT Fuze, M155A1*
Fig 4-19 on p D995 for *Nose(or Tail) Fuze, M907*
Bomb Proximity or VT Fuzes, such as shown in:
Fig 4-20 on p D996 for *VT Fuzes, Ring Type and Bar Type*
Fig 4-21 on p D997 for *Nose VT Fuze, AN-M166*
Bomb Hydrostatic Fuzes, such as shown in:
Fig 4-22 on p D99 & Fig 4-23 on p D1000 for *Tail Hydrostatic Fuze, AN-Mk230*
Bomb Nonstandardized Fuzes, such as shown in
Fig 4-24 on p D1001 for *Nose MT Fuze, M129*
Fig 4-25 on p D1002 for *Nose MT Fuze M130*
Fig 4-26 on p D1004 for *Nose MT Fuze, M131A1*
Bomb Pyrotechnic Fuzes, such as shown in:
Fig 4-27 on p D1006 for *Nose Fuze, AN-M146A1* and in
Fig 4-28 on p D1007 for its Arming Mechanism

Fuzes for Demolition Items
Fig 1-99a on p D926 is for Bullet Impact Fuze, M1A1 for Demolition Snake M3. It can also be used with "bangalore torpedoes", described in Vol 2 of Encycl, p B16-R

Fuzes for Hand Grenades:
Fig 1-100 on p D927 is for *Hand Grenade Fuze, M215*
Fig 1-101 on p D927 is for *Hand Grenade Fuze, M217*
Fig 1-102 on p D928 is for *Hand Grenade Fuze, M201A1*

Fuzes for Land Mines
Fig 1-103 on p D929 is for *Antipersonnel Mine, NM, M14 w/Integral Mine Fuze*
Fig 1-104 on p D930 is for *Antipersonnel Mine, M2A4 w/Mine Fuze, M6A1*

Fig 1-105 on p D931 is for *Antipersonnel Mine, M16 w/Combination Mine Fuze M605*
Fig 1-106 on p D931 is for *Antitank Mine Fuze, M603(T17E2)*
Fig 1-107 on p D932 is for *Heavy Antitank Mine, M6A2 w/Mine Fuze, M603*
Fig 1-108 on p D932 is for *Light, HE, Antitank Mine M7A2 w/Mine Fuze, M603*

Refs for Fuzes. See the following refs listed in Vol 4 of Encycl, pp D1025ff:
p D1025-L (Ref 17); p D1025-R (Ref 19); p D1025-R to D1026-L (Ref 23); D1026-R & D1027-L (Ref 27a); D1027-L (Ref 27b); D1027-L & R (Ref 28a); p D1027-R (Ref 28b); D1027-R & D1028-L (Ref 29); D1028-R (Ref 30a); D1028-R (Ref 30c); D1028-R (Ref 31); D1028-R to D1029-L & R (Ref 32); D1031-L (Ref 35); D1031-R (Ref 40); D1032-L (Ref 41); D1032-R (Ref 45); D1033-L (Ref 45e); D1034-R to D1035-R (Ref 51a); D1036-R to D1037-L (Ref 52); D1037-R (Ref 58)
Picatinny Arsenal Technical Reports on Fuzes are listed in Vol 4, pp D1057-R & D1058-R. Include also J.M. Kaschak, PATR **3377** (1966) (Production engineering activities relating to the M423 and XM427E1 fuzes)
Picatinny Arsenal Memorandum Reports on Fuzes are listed on pp D1058-R to D1560-L
Addnl Refs for Fuzes: 1) Berger, "Fuzes, Proximity, Radioelectric", MAF **25**, 77–89 (1951) 2) G.E. Grill & W. Rosell, "Fuze, Proximity", MAF **33**, 741–96 (1959) 3) P.I. Toropainen, "Verzögerungssatzstück fur Zünder (Distortion-free ring of fuze)", **DAS 1101250 Kle3**. Abstracted in Explosivst **10**, 83 (1962) 4) G.H. Hynard, "Mass Production of Fuzes", Ordnance **48**, 540–45 (1964) 5) Anon, Ammunition Series, Fuzes", **AMCP 706-210** (1969) 6) Military Handbook. "Fuze Catalog", **MIL-HNBK-137** (1970) 7) G. Cohn, "Sources for Fuze Information", Franklin Institute Research Labs, Paper Presented at AOA Fuze Meeting (Nov 15–16, 1972)

Fuzes and Fuze Components, Environmental and Performance Tests For. The following tests are described in **MIL-STD-331** (10 Jan 1966) and Change Notice 1 (24 July 1967); Change Notice 2 (11 December 1967); Notice 3 (11 June 1969); Notice 4 (16 April 1971); Notice 5 (1 June 1971); and Notice 6 (20 March 1972):
1. *Accidental Release (Low Altitude, Hard Surface), Test 206* is also briefly described in Vol 4 of Encycl, p D1092-L
2. *Air Delivery, Simulated (Parachute Drop) Test T213* is briefly described in Vol 4, p D2092-R and its revision is given as *Test 117* in Notice 3, 11 June 1969
3. *Catapult and Arrested Landing Test 212* is briefly described in Vol 4, p D1092-R
4. *Detonator Output Measurment by Lead Disc Test 302* is also briefly described in Vol 4, p D1092-R
5. *Dust Test 116.1* (Notice 5). A brief description of similar test, entitled "Sand and Dust Test" is given in Vol 4, p D1097-L. The "Dust Test" described in Notice 5 of 1971 is intended for use during development and production of fuzes to check their ability to withstand exposure to a fine dust environment. The test consists of placing bare live fuzes in a special chamber, provided with accessories to control dust concentration, air velocity, temperature and humidity. The free air space in the chamber must be sufficient to provide adequate circulation of the dust. Not over 15% of the cross-sectional area and 20% of the volume should be occupied by fuzes. The dust used in the test shall be of angular structure and consist of at least 97% silicon dioxide (SiO_2) and shall have the following size distribution as detd by wt using US Std Sieves: 100% pass thru 100-mesh, 98% thru 140, 90% thru 200 and 75% thru 325-mesh screen. Details of the test are given on three pages. It is time consuming because it consists of several phases: 1) Room Temperature Phase (duration not given); 2) Transition Phase (duration 16 hrs); 3) High Temperature Phase (duration 6 hrs). The fuzes must be safe and operable after these tests
6. *Explosive Component Output Measure-*

ment by Aluminum Dent Test 303 is briefly described in Vol 4, pp D1092-R & D1093-L

7. *Explosive Component Output Measurement by Steel Dent Test 301* is briefly described in Vol 4, p D1093-L and its revision *Test 301.1* in Change Notice 1, 24 July 1967

8. *Extreme Temperature Storage Test 112* is briefly described in Vol 4, p D1093-L

9. *Field Parachute Drop Test 211* is briefly described in Vol 4, pp D1093-L & D1093-R

10. *Five-Foot Drop Test 111* is briefly described in Vol 4, p D1093-R

11. *Forty-Foot Drop Test 103* is briefly described in Vol 4, p D1093-R

12. *Fungus Resistance Test 110* is briefly described in Vol 4, p D1094-L

13. *Impact Safe Distance (Projectile) Test 208* is briefly described in Vol 4, p D1094-L

14. *Jettison (Aircraft Safe Drop)(Fuzes) Test 201* is briefly described in Vol 4, p D1094-L, while its revision is given as *Test 201.1* in Change Notice 1 (24 July 1967)

15. *Jettison (Aircraft Safe Drop)(Fuze System) Test 205* is briefly described in Vol 4, p D1094-L & R

16. *Jettison Aircraft Safe Firing (Rocket Type) Test 204* is briefly described in Vol 4, p D1094-R

17. *Jettison (Simulated Aircraft Drop from Ground Launcher)Test 203* is briefly described in Vol 4, p D1094-R

18. *Jettison (Simulated Aircraft Safe Firing from Ground Launcher)(Rocket Type) Test 202* is briefly described in Vol 4, pp D1094-R & D1095-L, while its revision as *Test 202.1* is given in Change Notice 1, 24 July, 1967

19. *Jolt Test 101* is briefly described in Vol 4, p D1095-L, while its revision as *Test 101.1* is given in Notice 3 (11 June 1969)

20. *Jumble Test 102* is briefly described in Vol 4, p D1095-R, while its revision is given as *Test 102.1* in Notice 3 (11 June 1969)

21 *Leak Detection Test 118 (Notice 4)*, devised in 1971 was not described in Vol 4 of Encycl, published 2 years earlier. This test is applied to live fuzes filled with either halogen or helium to check the integrity for leaks that may affect safety and operability. Detailed description of equipment and test is given on six pages but with no illustrations

22. *Missile Pull-Off from Aircraft on Arrested Landing (Ground Launcher Simulated) Test 209* is briefly described in Vol 4, p D1095-R

23. *Muzzle Impact Safety (Projectile) Test 207* is briefly described in Vol 4, p D1096-L

24. *Rain Test (Exposed Fuze Storage) Test 109* is briefly described in Vol 4, p D1096-L

25. *Rough Handling (Packaged) Test 114* is briefly described in Vol 4, pp D1096-L & R

26. *Salt Spray (Fog) Test 107* is briefly described in Vol 4, pp D1096-R & D1097-L. Its revision given as *Test 107.1*, entitled *Salt Fog* is in Notice 6 (20 March 1972)

27. *Sand and Dust Method 116* is briefly described in Vol 4, p D1097-L. Its revision is given in Change Notice 1 (24 July 1967). See also *Dust Test 116.1*, Item 5 above

28. *Static Detonator Safety Test 115* is briefly described in Vol 4, p D1097-L & R

29. *Temperature Humidity Test 105* is briefly described in Vol 4, p D1097-R

30. *Thermal Shock Test 113* is briefly described in Vol 4, p D1097-R

31. *Time-to-Air Burst (Projectile Time) Test 210* is briefly described in Vol 4, p D1098-L

32. *Transportation Vibration Test 104* is briefly described in Vol 4, p D1098-L & R. Its revision in Change Notice 1 (24 July 1967) supersedes MIL-STD-331, p 1 of 10, January 1966 (8 pages)

33. *Vacuum-Steam Pressure Test 106* is briefly described in Vol 4, p D1098-R

34. *Waterproofness Test 108* is briefly described in Vol 4, p D1098-R

Besides the above tests there are about 90 tests for fuzes listed on pp D1098 to D1103-L and also 45 refs are listed on pp D1103-R to D1107-L of Vol 4 of this Encycl

Fuzes, Physical Testing. Eighty-nine tests are either briefly described or listed in Vol 4 of Encycl, pp D1099-L to D1103-R. There are forty-nine refs listed on pp D1103-R to D1107-L

Fuze, Sources for Information. A paper entitled "Sources for Fuze Information" was prepd in 1972 by Gunther Cohn, Senior Staff Engineer, Franklin Institute, Research Laboratories, Philadelphia, Pa, 19103. The price is $2.50, incldg postage

We are quoting its Abstract:

"The post-WorldWar II information explosion has left its mark on fuze documents as well. Now for the first time, here is a serious effort to bring together useful information in the field of fuzing. Containing 125 prime references with annotations and a score of sources, the paper has four categories:
(1) the general information needed to find one's way thru the fuze maze, including notes on fuze cstegories and reference availability,
(2) the basic references and agencies that serve as sources of fuze information,
(3) the document collections containing fuze information, like standards, engineering design handbooks and document centers, and
(4) references on specific topics of key interest"

Fuze, Spitback. A fuze located in a nose of a shaped-charge munition. When initiated by impact it produces a deton which is directed toward the base. The combination of Point Impact Fuze and base element is referred to as a PIBD (Point Initiating Base Detonating) Fuzing System
Ref: Glossary Ordn (1959, 128-R

Fuze Train, also known (when combined with a projectile charge) as "High-Explosive Train" or "Bursting Charge Explosive Train" Its definition is given in Vol 4 of Encycl, pp D837-R & D838 (See also Fig 1-21a on p D838-L & Fig 1-21b on p D839-L)

German Fuze Train, known as *Zündersatz* consisted practically of the same elements as American. Compositions of many of Ger Fuze Trains were detd during WWII at PicArsn and the results taken from PATR **1555**(1945), pp 11–15 are listed in PATR **2510**(1958) in Table 17 on p Ger 65

FZG. Same as German "Flying Bomb" V–1 (Vergeltungswaffe Eins) – "Revenge Weapon One", which is described in PATR **2510** (1958), p Ger 213-L and illustrated on p 214

G

"G" (Pólvora progresiva de fusil tipo "G"). A progressive rifle proplnt manufd after WWII at Asturias (Spain) by the Fábrica de Pólvoras y Explosivos. It consisted of NC 97.75, DPhA 0.75, Na oxalate 1.00, volatiles 0.40 and graphite 0.1% which, previous to coating the grains of proplnt, were slurried in 2 to 4% alcoholic soln of Centralite or camphor. This coating served to moderate the initial combustion and to render the proplnt progressive burning. Na oxalate served as a muzzle flash reducer and DPhA as a stabilizer. The grains were in the form of squares 1.20 to 1.50mm across and 0.25 to 0.35mm thick
Ref: Vivas, Feigenspan & Ladreda, Vol **3** (1948), 225

"G" (Pulver). See under Cool Propellants in Vol **3** of Encycl, p C511-R and also under Gallwitz in Vol **5**, p E116-L

"G1" (Pulver). Old Austrian Black Powder used in cannons. Type a (grains 7mm) was used in 80 x 90mm cannons, while Type b (grains 13mm) in cannons 120 & 150mm
Ref: Daniel (1902), 322

"G3" (Fusehead). See Fusehead "G3"

"G-117-b" (Rocket Powder). A double-base solventless extruded proplnt consisting of NC 50.0, NG 30.0, DNT 14.5, Ethyl Centralite 4.0 & K sulfate 1.5% with carbon black 0.02 & Pb stearate 0.40 added. This proplnt is smokeless, but has a high heat of expln which may give nozzle & trap erosion
Ref: W.E. Campbell Jr & L.H. Brown, Aerojet Report No **194**(1946), p 12

GA. Chemical Warfare Agent belonging to the group of *nerve gases*, known as *G-series*. It is one of the *G-Agents*. Its formula and brief description are given in Vol **2** of Encycl, p C167-R. It was first prepd during WWII in Germany by treating the dichloride of dimethylaminophosphoric acid (an irritating agent, known as *Product 39)* with Na cyanide, ethanol and chlorobenzene. It was known as **Tabun** or **Trilon 83**. It was used mixed in bombs and rockets with 20% chlorobenzene serving as a stabilizer, under the name **Tabun B**

In Glossary of Ordn (1959), p 130-L, GA is called *Ethylphosphorodimethylamidocyanide* (See also under G-Agent)

Gabeaud, Louis (1892–1951). A French ballistician, last holding the rank of Ingénieur Général des Fabrications d'Armament. Numerous publications from the year of 1921 in MAF (Mémorial de l'Artillerie Française), CR (Comptes Rendus de l'Académie des Sciences), Revue de l'Artillerie and Technique et Science Aéronautique
Ref: Anon, MAF **26**, 497–501 (1952) (Obituary)

Gacon. Same as Grenadine

Gadolin, A.W. (1828–1892). A Russian ballistician who, in collaboration with N.V. Mayevskii, proposed using propellant in hexagonal form with one or 7 perforations
Refs: 1) M.E. Serebryakoff, "Interior Ballistics of Gun Barrel Systems and of Solid Propellant Rockets", Russian Book published by Oboronghiz in 1962. Abbr translation by Dr V. Nekrassoff available in PicArsn Library 2) A.G. Gorst, "Powders and Explosives", Mashinostroyeniye, Moscow (1972), p 9 (in Russian)

Gadolinium, Gd. Rare-earth element having atomic No 64 & atomic wt 157.26; lustrous metal, sp gr 7.87; mp 1350° (Lange), 1325° (Ref 2); bp 2725° (approx); sol in dil acids, reacts slowly with w. Exhibits a high degree of magnetism, especially at low temp. Was first obtd in 1880 by Marignac from *gadolinite*,

which is a natural silicate of beryllium, iron and yttrium with rare-earth metals. Crystalline compds of Gd, especially, $Gd_2(SO_4)_3.8H_2O$ are used in magnetic method of obtg extremely low temps. Gd has a high thermal neutron capture cross-section and is used as an alloy in stainless steel for nuclear control. Some of its salts, as well as those of germanium and gold, were patented by DeMent in smoke producing compns (Ref 3)
Refs: 1) Hackh's Dict (1944), 364-L
2) CondChemDict (1961), 524-L 3) J. DeMent, USP 2995526(1961) & CA **55**, 25100 (1961)

Gadolinium Oxide, Gd_2O_3, mw 362.52; wh to cream-colored powder, sp gr 7.407 at 15/4°, mp 2330°; hygroscopic and absorbing CO_2 from the air; insol in w; sol in acids except HF. Used in nuclear reactor control rods, neutron shields, catalysts, dielectric ceramics, filament coatings, special glasses and as P activator
Refs: 1) Hackh's Dict (1944), 364-L
2) CondChemDict (1961), 524-R

Gaens Explosive, patented in 1889 in Germany, was prepd by mixing collodion cotton (gelatinized by ethyl acetate) with saltpeter and Amm ulmate. The latter compd is the salt of ulmic (or geic) acid, $C_{20}H_{14}O_6$, which was obtd from peat, previously washed with a soln of Na carbonate
Refs: 1) Daniel (1902), 322 2) Hackh's Dict (1944), 878-L

"GAF" Carbonyl Iron Powders. Trademark of General Aniline and Film Corp, 435 Hudson St, New York, 10014, for microscopic almost perfect spheres of very pure iron (99.6–99.9% Fe). They are produced in eleven carefully controlled grades ranging in particle size from 3 to 20 microns in diam. Used in high frequency cores for radio, telephone, television, short wave transmitters, radar receivers and direction finders. Also used as alloying agents, catalysts, in powder metallurgy and in magnetic fluids
Ref: CondChemDict (1961), 524-R

G-Agent. Any of a group of war gases, known as *nerve gases.* This group is called **G-Series** (Ref 2)

The following nerve gases are described in Vol 2 of Encycl (Ref 3), under CHEMICAL AGENTS: **GA or Tabun** (p C167-R); **GB or Sarin** (p C167-R); **GD or Soman** (p C168-L) and **GF** (p C168-L)

More detailed description is given for three of the above, originally German, gases in PATR **2510** (Ref 1), under Trilons on p Ger 204-L: **Tabun** (Trilon 83, T83 or T100); **Sarin** (Trilon 46, T46 or T114) and **Soman** (Trilon ?)
Refs: 1) Fedoroff et al, PATR **2510**(1958), p Ger 204-L 2) Glossary of Ord (1959), 131-R 3) Fedoroff & Sheffield, PATR **2700,** Vol **2**, pp C167-R to C168-L

GAGES (Brit GAUGES). There are several meanings for the term "gage", which include: **Gage Measures** (such as "wire gage", "sheet metal gage", "firearms gage", etc) and **Instruments for/or Means of Measuring or Testing,** (such as "Bichel Gage", "Copper Crusher Gage", "Diaphragm Gage", "Piezoelectric Gage", "Strain Gage", etc)
Gage of Firearms. In case of a shotgun, the gage is expressed as the number of lead balls of the diameter of the bore required to weigh 1 lb. Thus a 12 gage shotgun has a bore diameter of 0.729 inch because 12 lead balls of 0.729 inch diam weigh 1 lb

The measurements of shotguns of current sizes are standardized on the above basis as follows: 8 gage=0.835 inch; 10 gage=0.775, 12 gage=0.729, 14 gage=0.693, 16 gage=0.662 and 20 gage=0.615 inch (Ref 1, p 64 & Ref 2, p 131)

The following gages for measuring pressure developed by expls and proplnts are described in Vol **3** of Encyclopedia (Ref 3): *Bichel Pressure Gage* or *Bichel Closed Bomb* (pp C331-R & C332-L)

British Service Closed Vessel Gauge (pp C333-R & C334-L)
British Service Crusher Gauge (p C336)
British Strain Gauge (p C341)
Burlot-Malsallez Gage (French) (p C332)
CSE (Commission des Substances Explosives) *Closed Bomb Gage* (French) (p C332-R)
Dolgov Bomb Gage (Russian). Similar to Bichel Bomb but arranged vertically (p C331-R)
Fluid Pressure Cell SR-4 of Baldwin (US) (p C342-R)
German Closed Bomb Gages (p C333-R)
Kistler Quartz Pressure Transducer (US) (p C342-R)
Optical Spring Gauge. See Petavel Manometer
Petavel Manometer (British) (p C337-R)
Piezoelectric Gage (Quartz) (US) (p C339-R)
Piezoelectric Gage (Tourmaline) (US) (p C340-R)
Sarrau-Vieille Closed Bomb Gage (French) (p C333-L)
Spring or Mechanical Gauges (British), which include Petavel Manometer (See above) and *High-Pressure Spring Gauge* (p C338)
US Closed Vessel Gages. One of them, presented in Fig on p C334, is described on p C335. The so-called *External Crusher Gage T14* is shown in Fig on p C336-L, while the *Internal Crusher Gage M11* is in Fig on p C337-L
US Strain Resistance Wire Transducer Pressure Gage, Model C-AN is described on p C342 and illustrated in Fig on p C341-R
Vieille Type Bombs (French) are mentioned on pp C331-R & C332-L
Refs: 1) Ohart (1946), p 64 2) Glossary of Ordn (1959), pp 130ff 3) Fedoroff & Sheffield, Encycl of Expls, Vol **3**, pp C331 to C343 (1966)

Gages, Crusher. See above under GAGES

Gages, Diaphragm. See Fig G1. The gage is placed a fixed distance from the expl to be tested and the deflection of the steel or copper plate disc is measured. Because of the complex nature of the response the gage is useful only for comparison purposes
Refs: 1) P.M. Frye & J.E. Eldridge, OSRD **6248** (1946) 2) R.H. Cole, "Underwater Explosions", Princeton Univ Press, Princeton, NJ (1948), pp 157–58

Gages, Optical Spring. See above, under GAGES

Gages, Piezoelectric. See above, under GAGES

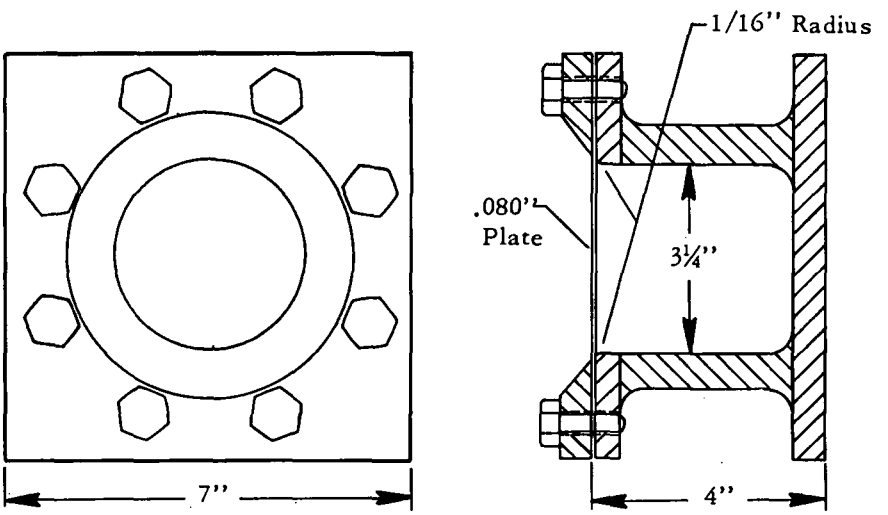


Fig G1 Construction of UERL diaphragm gauge.

Gages, Spring. See above, under GAGES

Gages, Strain. The fundamental idea of a strain gage is that stretching or compressing a length of wire will change its resistance. The length of wire on stretching increases, while at the same time the diam decreases, and since resistance varies directly as the length and inversely as the diam, these both effects act to increase the resistance. Then, if such a wire is properly attached to a member which is to be stressed, then the resulting deformation (strain) will be transmitted to the wire changing its resistance. Knowing the character of the stress and the dimensions and material of the stressed member, the change in gage resistance can be related to the dimensional change or strain in the stressed member. Fig G2 shows the construction of a typical size SR-4 strain gage made by Baldwin Lima Hamilton Corp

In addition to changes in dimension of the gage wire, there is also a change in metal properties of the wire. In an alloy known as Advance (55% Cu, 45% Ni) a change in length of 0.1% causes a change in resistance of about 0.2%. The ratio of percent resistance change to percent length change is known as the "gage factor". Gage wire is usually 0.001 inch in diam and, except in certain types, is embedded in a suitable cement and mounted on a thin piece of paper. To use a gage as shown in Fig G2, it is attached in the desired spot on the stressed member with nitrocellulose cement (Duco cement, Baldwin SR-4, or equal). The cement penetrates the paper mounting and forms a bond between the wires and the test object. The gage and the cement thus undergo the same tensile or compressive stress as the member they are attached to. When there are stresses acting in more than one direction, a special type of gage is used as shown in Fig G3. These gages are called rosettes. Gages can be operated with nitrocellulose cement up to 180°F. The wires are embedded in Bakelite plastic and applied with a Bakelite cement for use from 180°F to 400°F. Above 400°F ceramic cement is used. Fig G4 is a special type of unbonded gage which is used between a fixed frame and a movable armature

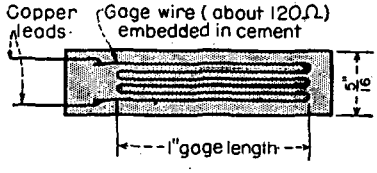


FIG G2 Construction of a typical size of SR-4 bonded strain gage

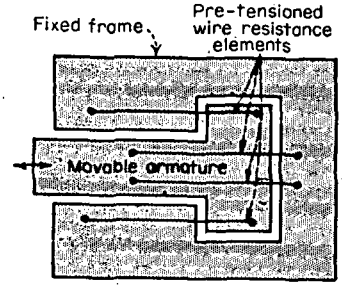


FIG G4 Simple form of unbonded strain gage developed by Statham Laboratories

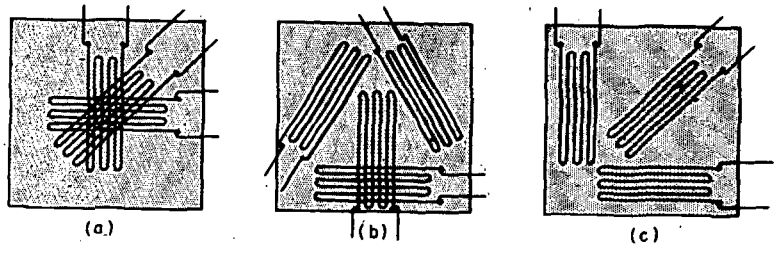
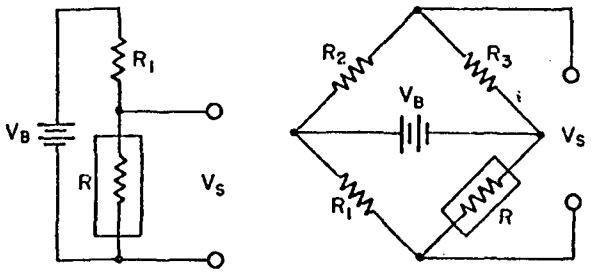


FIG G3 Three designs of bonded rosettes used where stresses are multi-directional



(A) BALLAST CIRCUIT (B) BRIDGE CIRCUIT

FIG G5 Simplified Strain Gage Circuits

Elementary strain gage circuits are shown in Fig G5. The first circuit [Fig G5(A)] is probably the most simple that may be used with a strain gage. For this circuit the signal voltage may be represented by:

$$V_s = V_b\left(\frac{R+\Delta R}{R+\Delta R+R_1} - \frac{R}{R+R_1}\right)$$

where: V_b = signal voltage due to the strain, volt
V_s = battery voltage, volt
R_1 = ballast resistor, ohm
R = Strain gage resistance when relaxed, ohm
ΔR = change in strain gage resistance due to strain, ohm

The two terms in the parentheses of this equation represent the potential in the unstrained condition (the right term) and in the strained condition (the left term). By setting the value of the ballast resistor R_1 equal to the strain gage resistance R, a process that is often used in practice, the equation for the signal voltage becomes:

$$V_s = \frac{\epsilon F_g V_b}{2(2+\epsilon F_g)}$$

where: F_g = gage factor, dimensionless
ϵ = strain, in/in

The simple bridge circuit [Fig G5(B)] effectively eliminates the DC component of signal that was present in the ballast circuit. Except for the limination of the DC component, the magnitude of the signal voltage is identical to that of the ballast circuit provided the resistances R, R_1, R_2 and R_3 are equal. The battery voltage is usually 3 to 6 volts. A typical commercial application is shown in Fig G6 in which gages on loading screws show pressure between calendar rolls

Besides strain measurement, strain gages are used in measurement andcontrol, Load cells are prefabricated transducers and are increasingly used because the strain gages are already mounted. One application of a load cell is the rapid weighing of heavy equipment with infinitesimal motion

In military applications, strain gages are used to measure pressure in rifles and gun tubes, pyrotechnic ejection systems, gas generators and propulsion systems. See also US Strain Resistance Wire Transducer Pressure Gage under GAGES and in Vol 3 of Encycl, pp C341-R and C342

Refs: 1) R.W. Nolan, "Strain Gages", ChemEng **60** (No 9) (Sept 1953), pp 217–28
2) Anon, "Engineering Design Handbook. Military Pyrotechnic Series. Part IV. Design of Ammunition for Pyrotechnic Effects" US Army Materiel Pamphlet **AMCP 706–188** (1973), Sec 5-8

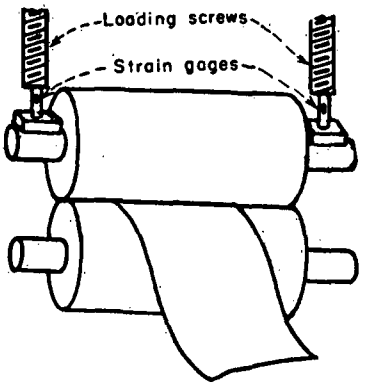


FIG G6 Strain gages on loading screws show pressure between calender rolls

Gain Rifling, Gain Twist, Gaining Twist, Progressive Rifling or Increasing Twist of Rifling (Ger names: Zunehmender- oder Wachsender Drall) . Rifling in which the degree of twist (turn) increases from the origin of rifling to the muzzle

Under the term of **rifling** are known helical *grooves* cut in the bore of a rifled gun tube beginning at the front face of the gun chamber (origin of rifling) and extending to the muzzle. The purpose of rifling is to impart spin and stability to the projectile, so that it will travel nose first to the garget. Engagement of projectiles greater than about .60 inch in diam with the rifling is generally accomplished by use of a *rotating band*, also known as *driving band* (See in Vol **5** of this Encycl

Accdg to Mr R.P. Baumann of PicArsn, Germans used during WWII progressive rifling in at least 13 of their guns. They are listed in Ref 2, p Ger 138-R

Some Amer weapons using "gain rifling" are listed by Mathews (Ref 1)

Refs: 1) J.H. Mathews, Ordn **42**, 62–5, July-Aug 1957 2) PATR **2510**(1958), p Ger 138-R 3) Glossary of Ordn (1959), p 306-R (Twist, increasing); p 245-L (Rifling)

Gaine *(Sheath).* A Brit term for a metallic cup (sheath) attached to the bottom of the fuze. When filled with a comparatively sensitive HE (such as Tetryl), then connected to a detonator and inserted into a cavity in the main expl charge of a projectile (known as *burster*), it performs the same function as a *booster*, described in Vol **2** of Encycl (Ref 3, pp B243 to B246-L)

The idea of separating, by a metallic sheath, the bursting charge of a projectile from the booster belongs to Sir R. Robertson and dates to the time of WWI. It was done in order to eliminate the possibility of premature explns caused by set-back. In its original form, the device was intended to detonate an insensitive expl such as Amatol. It consisted of a strong steel tube, 3 inches long and ¼ inch thick, screwed into the mouth of the shell and closed at the lower end with a plate strong enough to withstand the shock produced by a set-back and weak enough to be blown out by the expl wave of the booster. At the top of the tube there was a pellet of Black Powder with an axial perforation, which was ignited by the flash from the fuze screwed in over the gaine. The flash from Black Powder was passed to an open capsule containing Mercury Fulminate situated immediately over two pellets of Tetryl located in the gaine. The gaine was placed in the long cavity, located in a cylinder of cast TNT imbedded in Amatol, which was the main charge in the upper part of the shell. The bottom of the TNT cavity contained a bag filled with crystals of TNT. The Fulminate detonated the Tetryl pellets from which the detong wave passed thru the TNT crystals to the cast TNT and so on, with increasing velocity to the main charge of Amatol (Ref 1, pp 162–63)

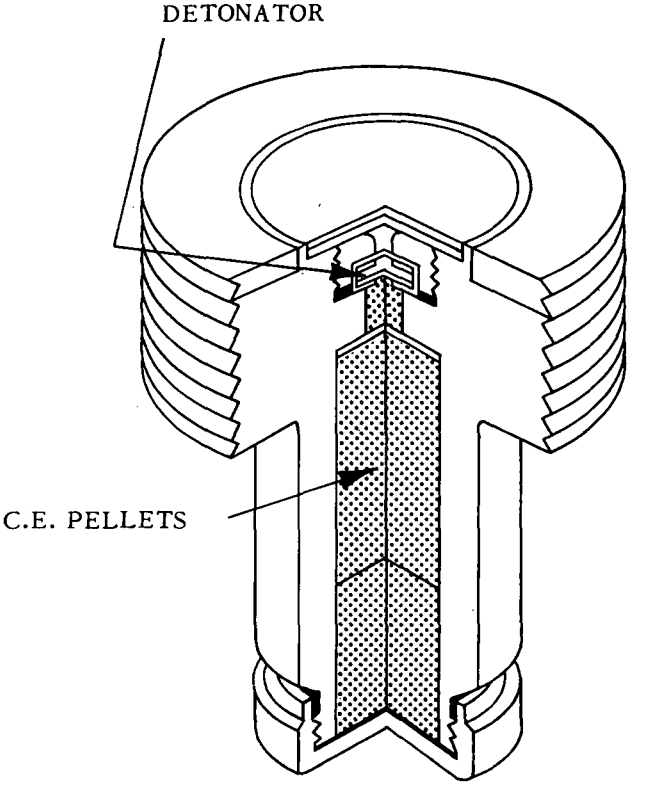


Fig G7 GAINE NO 13

under "Aldonic Acids, Their Derivatives and Nitric Esters"

d-Galactose and Derivatives

d-Galactose or Hexose, $C_5H_{11}O_5.CHO$; mw 180.16; wh prismatic crysts, mp 165.5°(for anhydrous) or 118–120° for its monohydrate; soly of anhydrous compd in w is 10.3% at 0°C and 68.3% at 25°); soly in alc – 0.6% at 38.5° in 85% alc; sol in pyridine; sl sol in glycerine. Can be derived from milk sugar by hydrolysis caused either by fermentation or by mineral acids.

On being nitrated it gives expl Pentanites, alpha and beta

Refs: 1) Beil **1**, 911 & (457) 2) F. Soxhlet, JPrCh [2]**21**, 269(1880) 3) CondChemDict (1961, p 524-R

d-Galactose Pentanitrate, $C_6H_7O(ONO_2)_5$; mw 405.14, OB +2.0%, N 17.29% (theory); N 17.18% (found). Nitration of d-galactose by slowly adding concd sulfuric acid to a soln of d-galactose in concd nitric acid (as indicated in Refs 1 & 2) produced (after crystn from alc) colorless, transparent ndls which melted at 115–116° and exploded above 126°. They were insol in w and sl sol in alc and reduced Fehling's soln slowly on warming. This compd was designated as *alpha-Galactose Pentanitrate.* The *beta-Galactose Pentanitrate* was obtd by evaporating alc mother liquor of alpha compd. It was in the form of transparent monoclinic ndls, melting at 72–73° and decompg at 125°. They were insol in w and very sol in alc; reduced Fehling's soln and decompd slowly when stored at 50°(Ref 3)

Refs: 1) Beil **1**, 915 2) W. Will & F. Lenze, Ber **31**, 74–5(1898) 3) Davis (1943), 243

Galcit (Abbr for Guggenheim Aeronautical Laboratory, California Institute of Technology). A brief description was given in Vol **1** of Encycl, p A497-R under *Asphalt-Perchlorate Castable Propellants.* The following may be added:

Galcits were heterogeneous mixts contg ca 25% of asphalt oil fuels and ca 75% of perchlorate oxidizers. Their specific impulse (I_{sp}) was ca 185 secs vs 200 for Ballistite

Refs: 1) Anon, "Elements of Armament Engineering", USMA (US Military Academy), West Point, NY(1954), p 39 2) Rocket Encycl (1959), p 181

Galena *(Galenite or Lead Glance),* PbS, mw 239.28; gr cubic crysts, sp gr 7.5, hardness 2.5, mp 1120°; insol in w & alkalies; sol in concd nitric acid and in hot hydrochloric. Occurs in many US States, Canada, So America, Africa, etc. Serves as a chief ore of lead

Refs: 1) Hackh's Dict (1944), 365-L 2) CondChemDict (1961), 525-L

Galeries d'essais. French Testing Galeries are briefly described in Vol **3** of Encycl, p C371, item D, under "Coal Mining Explosives, Testing for Permissibility"

Galette. French for **Cake,** such as used during manuf of proplnts. It is described on p C3 in Vol **2** of Encycl

Slightly different information was given by US Army Attaché Report of May 13, 1954, namely:

Galette 1–SD: NC 70.25–71.00 & NG 29.75–29.00% with water less than 1%

Galette 2–SD: NC 73.06 & NG 26.94% with water less than 1%

Note: SD stands for "sans dissolvant", which means "solventless'

Galex. Trade name of G & A Laboratories, Inc, Box 1217, Savannah, Georgia and of National Rosin Oil Products, Inc, 1270 Ave of the Americas, NY 10020 for a stable non-oxidizing rosin consisting principally of dehydroabietic acid; lt amber solid, sp gr 1.082 at 20/4°, softening p (B&R Method) 66°, fl p (Cleveland open cup) 210°C, fire

British gaines of WWII are described in Ref 2, which used to be confidential, and were not discussed by us earlier

The term gaine was also applied to Japanese boosters and two of such Army devices are illustrated in Figs on p B246 of Ref 3

British Fuze No 700 Mk II employs Gaine No 13, shown in Fig G7, for a rocket nose fuze. Expln of the gunpowder chge in the base of the fuze initiates the gaine which is located in the adapter ring below the fuze. The Nose Fuze No 731 Mk I also employs a gaine (not shown) used in rockets

Refs: 1) Marshall **3**(1932), 162–63 2) Anon, "British Explosive Ordnance", **TM9-1985-1** (1952), pp 352 & 365 3) Fedoroff & Sheffield, Encycl of Expls, Vol **2** (1962), pp B243 to B246, under BOOSTER

Gaine-relais. Fr for Adapter-Booster

gal. Abbrn for gallon. One US gallon = 4 quarts = 8 pints = 3.785332 liters = 0.833 Imperial gallon. One Imp gallon = 4.54596 liters ω 1.20032 US gallons.

d-Galactan and Derivatives

Galactan, Gelose, Galactosan or Carraghan Moss, $(C_6H_{10}O_5)_n$, mw $(162.14)_x$; white carbohydrate, giving on hydrolysis galactose, $C_6H_{12}O_6$.

A method of prepn from pectin matter of seeds of white lupine was described by Hirst et al (Ref 2). Vinogradova et al (Ref 3) prepd snow-white powder from same source contg 94.5% galactan, the rest being pentosan and uronic acids. Can also be obtd from agar-agar. Yields mucic acid on oxidation. Its aqueous solns were used by the Germans in some AN expls for controlling plasticity, such as in *Wetter-Wasagit B*, which contd: NG 27.8, NC 0.7, AN 30.5, NaCl 39.5, gelose 0.7, woodmeal 0.3 & talc 0.5% (Ref 4)

Refs: 1) Beil, not found 2) E. L. Hirst et al, JCS **1947**, 1225–29 & CA **42**, 1203d (1948) 3) L.M. Vinogradova et al, ZhurPrikladnoyKhim **27**, 1302–06(1954) & CA **49**, 9269(1955)(Engl transln 62G6R, which may be obtd from Associated Technical Services, PO Box 271, East Orange, NJ) 4) PATR **2510**(1958), p Ger 68-R (Gelose)

d-Galactan Trinitrate or Galactasan Trinitrate, $[C_6H_7O_2(ONO_2)_3]_x$; mw $(297.14)_x$, N 14.14%, OB to CO_2 −24.2%; white crysts, sl sol in acet. Accdg to Davis (Ref 2), it was obtd by the action (during several days) of mixed nitric-sulfuric acid on d-galactose and then deposited from alcohol in crusts of small crysts. Vinogradova et al (Ref 3) prepd it by nitrating galactan of 94.5% purity; the resulting product contg 14.03% nitrogen, approx corresponded to Galactan Trinitrate. Nitration was conducted by means of an anhydrous mixture of nitric acid 45, phosphoric acid 45 and phosphoric anhydride 10% at 0°C

It was an expl which was unstable, probably due to the fact that it was not pure. Its sensitivity to impact was comparable to that of Tetryl and its brisance and power were slightly lower than of TNT

Refs: 1) Beil, not found 2) Davis (1943), 243 3) L.M. Vinogradova, ZhurPriklKhimii **27**, 1302–06(1954)(See Ref 3, under Galactan)

d-Galactonamide and Its Pentanitrate are briefly described on pp A122-R & A123-L in Vol 1 of Encycl, under "Aldonic Acids, Their Derivatives and Nitric Esters"

d-Galactonic Acid and Its Pentanitrate are briefly described on pp A122-R & A123-L in Vol 1 of Encycl, under "Aldonic Acids, Their Derivatives and Nitric Esters"

d-Galactonic Acid Pentanitrate, Methyl Ester or Methyl-d-galactonate Pentanitrate is briefly described on p A123-L in Vol 1,

p (Cleveland open cup) 240°; sol in ordinary org solvents, waxes and compatible with SR-S, Neoprene, rubber and many resins. Used for rubber-based pressure sensitive adhesives and for many other purposes which are indicated in Ref
Ref: CondChemDict (1961), 525-L

Galil Rifle. An Israeli weapon system that is not merely a rifle nor a light machine gun nor even a launcher for grenades, but a combination of all three. The Galil resembles the old German MP-44 that fired the 7.92mm short (Kurtz) cartridge so effectively in WWII. Like the MP-44, the Galil is heavier than its competitors (such as the Israeli New Uzi, the US M16, German Heckler & Koch 5.56mm, the Stoner 63, the AR-18, the Bretta & others) and fires more slowly. The Galil weighs less than 9 lbs without any magazine and fires at a cyclic rate of ca 60 rounds/minute

There are three different Galil magazines holding 12, 35 and 50 rounds, respectively. The Galil can deliver bullets & antipersonnel grenades as part of its basic functions. It can be used to discharge antitank (Energa) grenades which have more power, but less range than those from the US LAW (M72). It can also deliver high-angle mortar-type bombs comparable with those from 2-inch (52mm) mortars. Within the capabilities of the 5.56mm cartridge, the Galil appears to have a bright future. The Israeli Defense Industries is quoting a price of $150 for an Israeli-made Galil
Ref: J. Weller, National Defense (formerly Ordnance) **58**, No 320, 142–45 (1973)

Gallacetophenone and Derivatives
Gallacetophenone or *4-Acetyl-pyrogallol*, $C_6H_2(OH)_3COCH_3$; mw 168.15, ndls or plts (w), mp 173°; sol in alc & hot w. Prepd by heating at 145–50° pyrogallol, HAc & zinc chloride. **Picrate**, orn-yel ndls, mp 133°
Refs: 1) Beil **8**, 393, (685) & [439]
2) M. Nencki & N. Sieber, JPrCh [2] **23**, 151, 538 (1881)

Gallacetophenone Azide or **Azidogallacetophenone** (w-Azido-2,3,4-trioxy-acetophenon, in Ger), $C_6H_2(OH)_3COCH_2N_3$; mw 209.16, N 20.10%, plates (xylene), mp 155°. Prepd by treating the w-chloro compd with NaN_3 in hot water
Refs: 1) Beil **8**, (686) 2) G. Barger & A.J. Ewins, JCS **97**, 2260 (1910) & CA **5**, 1083 (1911)

Gallaher Powder. A modification of BkPdr contg, besides saltpeter carbon & sulfur, the sulfates of Fe & Cu and pulverized tan bark. It was patented in the US by Lloyd & Walker
Ref: Daniel, Dict (1902), 322

Galleries for Testing Coal Mining Explosives and Gallery Tests. See in Vol 2 of Encycl, pp C368-R to C378-L, under "Coal Mining Explosives, Testing for Permissibility" and also *Versuchstrecke Dortmund-Derne* (Testing Gallery at Dortmund-Derne, Germany) in PATR **2510** (1958), p Ger 215-R

Gallery Practice Ammunition. Small Arms ammunition with a reduced charge used in gallery practice and also for guard purposes
Ref: Glossary of Ordn (1959), p 133-L

Gallic Acid or **3,4,5-Trihydroxybenzoic Acid**, $(HO)_3.C_6H_2.CO_2H.H_2$; mw 188.13; colorless or sl yel ndls or prisms; sp gr 1.694 at 4/4°, mp loses water at 100°, decomp at 235°; sl sol in w or in eth; sol in alc or in glycerin. Can be obtd by the action of mold on solns of tannin (gallotannic acid) or by boiling the latter with strong acid or caustic soda. Used as a reagent for detecting small amts of ferric salts and acids; also in manuf of inks, pyrogallol. Davis (Ref 2) describes its use in mixtures with $KClO_3$ in Non-Picrate Whistlers

Its US specification requirements and tests are in Ref 5

Refs: 1) Beil **10**, 470, (236) & [335] 2) Davis (1943), p 73 (Use in fireworks) 3) Hackh's Dict (1944), 365-R 4) Cond-ChemDict (1961), p 525-L & R 5) US Specification MIL-A-13601 (Ord) (Cancelled and not in files of Picatinny Arsenal)

Gallic Powder, patented in 1869 by Horsley, England, contd K chlorate 75 & galls or nutgulls (noix de galle) 25%
Ref: Daniel (1902), p 322

Gallium (Ga) or *Austrium*, Ga, atomic wt 69.72, atomic No 31; silvery-wh metal, sp gr 5.885 at 24°, mp 29.7°; bp 1600°(Ref 2), 2535° (Lange); insol in w; sol in acids & alkalies; sl sol in Hg. Obtd from bauxite and Zn ores. Has been suggested for use as a backing material for optical mirrors and as a possible heat exchange medium in nuclear power reactors
Refs: 1) Hackh's Dict (1944), 365-R 2) CondChemDict (1961), 525-R

Gallium Triazide, $Ga(N_3)_3$. See Vol **1** of Encycl, p A536-L

Gallotannic or **Tannic Acid** (Digallic Acid or Tannin), $(HO)_3C_6H_2.CO_2.C_6H_2(OH_2.CO_2H$; mw 322.22, sl yel amor powder, mp decompd 200–210°; sol in w, alc & acet; almost insol in eth, benz, chlf & petr eth. Naturally occurring, probably as a glucoside, in gall-nuts (nutgulls) and in tree barks (such as sumac, oak & hemlock). Used for tanning skins, prepn of chemicals (such as gallic acid, pyrogallic acid and hydrosols of noble metals). Also used as a component of some expls such as French Poudre de mine de sûreté, described in Daniel, Dict (1902), p 761 and for treatment of skin burns
Refs: 1) Hackh's Dict (1944), 829-R (Tannic Acid) 2) CondChemDict (1961), 1105

Galls (Nutgalls, Aleppogalls, or Turkey galls). Excrescences on various kinds of oak trees resulting from the deposition of insect eggs. The best grades (55–60% gallotannic acid) come from Iran, Syria, Turkey and Tripoli; the poorer grades from Italy, France, Germany and Austria. Galls come in powders of colors ranging from black thru green to white, owing to degrees of maturity; the darker being more mature and contg more gallotannic acid. Serves as source of gallic & gallotannic acid, manuf of inks and as a component of some older types of expls, such as Gallic Powder (qv)
Ref: CondChemDict (1961), 525-R & 526-L

Gallwitz, Uto (? –1943). Ger General, one of the most prominent ordnance specialists. He introduced before WWII the low calorific value propellants (such as **"G" Pulver**) by substituting DEGDN & TEGDN for NG. He also introduced NGu (Nitroguanidine) as a cooling agent and this proplnt became known as **Gudolpulver**

Gen Gallwitz was killed during WWII at the Russian Front

His work on the development of cool proplnts is described in the book: Uto Gallwitz. "Die Geschützladung", Heereswaffenamt, Berlin (1944), which was classified during WWII in Germany as "secrete"

See also Cool (Cooled) Propellants in Vol **3** of Encycl, p C511-R to C512-L

Galvani, Luigi (1737–1798). An Italian physician and anatomist who discovered the galvanic current in the course of working with frog muscles
Refs: 1) Hackh's Dict (1944), 366-R 2) EncyclBrit **9** (1964), p 1107

Galvanic (Voltaic). Pertaining to an electric current produced by chemical action
Ref: Hackh's Dict (1944), 366-R

Galvanism. A branch of physics which deals with electric currents produced by chemical action, as opposed to those produced by heat, friction or induction
Ref: Hackh's Dict (1944), 366-R

Galvanize. To protect an easily oxidizable metal (such as iron) with a less oxidizable metal (such as tin or zinc) by electric means
Ref: Hackh's Dict (1944), 366-R

Galvanograph. The photographic record of a sensitive galvanometer arranged so that the mirror deflects a beam of light onto a moving film or paper
Ref: Hackh's Dict (1944), 366-R and Fig on p 367-L

Galvanometer. An instrument for detecting and measuring the strength of an electric current. It consists essentially of a magnetic needle suspended in a wire coil. The slightest deflection of the needle produced by a current thru the coil is measured by some optical system, eg reflection of a beam of light from a small mirror attached to the needle, and observation of the reflected beam on a scale. The coil may be fixed and the magnet movable or vice versa
Ref: Hackh's Dict (1944), 366-R & 367-L

Galvanometer Chronograph was invented in 1849 by Pouillet and in 1851 by Helmholtz. It was improved by Hamburger (in 1885), Ramsauer (1903) and Timme (1921)
Ref: Cranz, Ballistik, Vol 3(1927), 101–07
Note: Other types of chronographs are described in Vol 3 of Encycl, pp C307-R to C311-R

Galvanometric Titration Method. As exemplified by the Karl Fischer Method, the KF Reagent is added in excess to the test solution, and the excess back titrated with aqueous methanol. If a pair of Pt electrodes be immersed in the soln, connected to a power source and a galvanometer, then the latter will show a current so long as any iodine (which is what is being back titrated) is left. See also under Karl Fischer Method on pp D1622-L to D1628-L in Vol 5 of Encycl
Refs: 1) E. Salomon, ZPhysikChem **24**, 55 (1897) and **25**, 366(1898) 2) G. Wernimont & F.J. Hopkinson, IndEngChem(Anal Ed) **12**, 308–10(1940) 3) K.G. Stone & H.G. Scholten, AnalChem **24**, 671–674(1952)

Gambir or Gambier (Pale Catechu). An odorless brown powder prepd by drying an extract from a decoction of the leaves and twigs of an Indian shrub, *Ourouparia or Uncaria gambir.* It is insol in w and sol in alc. Used as an astringent and tanning material
Ref: Hackh's Dict (1944), 367-R
Note: Someone has reported that gambir was used in admixture with K chlorate in some "safety explosives"

Gamma (γ) Cellulose. See Vol 2 of Encycl, under CELLULOSE AND DERIVATIVES, p C96-L. Alpha (α) and beta (β) celluloses are described on the same page

More detailed description is given in Ott, Vol 2, pt 1 (1954), p 12

Gamma Radiation. The combined process of emission, transmission and absorption of gamma rays, as from the expln of atomic bomb
Ref: Glossary of Ordn (1959), 133-L

Gamma Radiation Effect on Explosives. Using Au-198 radiation the most affected were NG and PbN_8, while TNT and Pb styphnate were most stable. Binary expls such as Ballistite & Comp B, primers such as M26 also were studied (Ref 1)

The effects of gamma radiation on four selected fluoroexpls:

MFTNB or PF	1-Monofluoro-2,4,6-trinitrobenzene or Picryl Fluoride
DFTNB	1,3-Difluoro-2,4,6-trinitrobenzene
TNTF	2,4,6-Trinitrobenzotrifluoride
TFEt Tetryl	2,4,6-Trinitrophenyl-β,β,β-trifluoroethylnitramine

are reported by Avrami et al (Ref 2). Their behavior at different exposure levels is compared with the behavior of the parent compds, TNB & TNT; RDX & HMX are also included as controls. The determinations made on each expl were wt loss, vacuum stability, mp, IR spectra, DTA, TGA, impact sensitivity, expln temp, and rate of detonation as a function of total gamma exposure. A brief description of the expln-containment irradiation capsule designed & used in these studies is also given

Avrami et al (Ref 3) also subjected a group of eleven expl materials in powder & pellet form to Co^{60} gamma radiation. Based on this work, a damage threshold as a function of total gamma dose was determined for each expl. The results indicate that the capability of the expls to withstand Co^{60} gamma radiation decreases in the following order: TACOT, TATB, DATB, HMX, Tetryl, TNB, TNT, RDX, PETN, NGu & BaN_6

Abbrns: **DTA** – Differential Thermal Analysis; **TACOT** – Tetranitro-1,2,5,6-tetrazadibenzocyclo-octatetrene; **TATB** – Triaminotrinotrobenzene; **TGA** – Thermogravimetric Analysis; **DATB** – Diaminotrinitrobenzene; **NGu** – Nitroguanidine

Refs: 1) H. Rosenwasser, USAtomicEnergyComm **ORNL-1720** (1955) & CA **50**, 14229 (1956) 2) L. Avrami, H.J. Jackson & M.S. Kirshenbaum, "Effects of Gamma Radiation on Selected Fluoroexplosives", PATR **3942** (June 1970) 3) Ibid, "Radiation-Induced Changes in Explosive Materials", PATR **4602** (Dec 1973)

Gamma Rays. Electromagnetic radiation similar to X-rays but of shorter wave lengths. Gamma rays originate in the nucleus of an atom, whereas X-rays originate in the extranuclear structure. Gamma rays are emitted by radioactive substances (such as radium) as secondary radiation caused by beta (β) rays striking matter

Note: Beta rays consist of a stream of negatively charged particles (electrons) emitted from radioactive substances with the velocity of light (186000 miles/sec or 2.99×10^{10} cm/sec). There are also *alpha rays*, which consist of a stream of positively charged particles emitted from radioactive substances with a speed of 20,000 miles/sec (1.922×10^{9} cm/sec)

Refs: 1) Hackh's Dict (1944), 367-R 2) Glossary of Ordn (1959), 133-L 3) CondChemDict (1961), 526-R; 8th edit (1971), 410-L

Gamma Rays, Analysis. The technique of making radiographs with γ-rays (Gamma Rays Analysis) is fundamentally similar to radiography with X-rays. It consists in placing the object to be examined (such as cast metal, ammunition, etc) betw the source of gamma ray radiation (such as radium sulfate or mesothorium) and a photographic plate or film. Any cavity in the sample will be shown on the film (or plate) as a dark region. Radiographs made with γ-rays are called *gammagraphs*. This technique is easier to apply than X-ray technique and is less expensive

The first γ-ray photographs were obtd in 1925 by Lipon & Laborde and this technique was adopted in 1928 in Russia. The work started in the US in 1929 at the Naval Ordnance Research Laboratory (Compare with Betatron, described in Vol **2** of Encycl, pp B108 & B109)

Refs: 1) G.E. Doan, "Gamma Ray Radiographic Testing", JFranklinInst **216**, 183–216 (1933) (Review of earlier radium radiography) 2) H.R. Grand et al, PhysRev **47**, 782 (1935) (Gamma-ray from disintegration of beryllium by Deuterons and Protons) 3) K. Lark-Horowitz et al, PhysRev **48**, 100 (1935) (Gamma rays from nitrogen bombarded with Deuterons) 4) R.G. Herb et al, PhysRev **51**, 691–98 (1937) (Gamma rays from light

elements due to proton bombardment) 5) H.E. Davis, G.E. Troxell & C.T. Wiskosil, "The Testing and Inspection of Engineering Materials", McGraw-Hill, NY (1941), pp 207, 208 & 216 6) N.L. Mochel in ASTM "Symposium on Radiography", Philadelphia (1943), pp 48–64 and 163–75 7) A. St. John & H.R. Isenburger, "Industrial Radiography", J. Wiley, NY (1943) (Description of various methods of testing; included 1314 refs) 8) Addenda to "Industrial Radiography", by H.R. Isenburger, lithographed and copyrighted by St. John Laboratory, Califon, New Jersey (1945) (Includes bibliography from 1942 to 1945, up to Ref 1699) 9) Ditto, Second Supplement includes bibliography from 1945 to 1948 up to Ref 2511 10) M. Hetényi, "Handbook of Experimental Stress Analysis", J. Wiley, NY (1950), p 982 (Fairly comprehensive description of γ-rays technique is given) 11) K. Siegbahm, "Beta and Gamma Ray Spectroscopy", Interscience, NY (1955) 12) W.J. Price, "Nuclear Radiation Detection", McGraw-Hill, NY (1958) 13) K. Siegbahm, Ed, "Beta and Gamma Ray Spectroscopy", Interscience, NY (1965) 14) A.J. Ferguson, "Gamma Ray Spectroscopy", Amsterdam, North Holland Pub Co, NY, Wiley (1965) 15) Damiaan de Soete, " Gamma Ray Spectrometry", Brussels, Paleis der Academien (1967)

Gamma-Rays, Behavior of Propellants under was discussed by:
E. Piantanida & M. Piazzi in Chimica e l'Industria (Milano) **42**, 1238–42 (1960) & CA **55**, 20434 (1961)

Gamma-TNT or **2,4,5-Trinitrotoluene.** Found to the extent of about 2.9% in manuf of 2,4,6-TNT, the principal isomer found in quantity 95.5%. Two other isomers: beta- (or 2,3,4) and zeta- (or 2,3,6) are found in quantities 1.3% and 0.3%, respectively. Gamma TNT consists of yel crysts, sp gr 1.629 at 20/4°, mp 104° and bp – expl at 290–91°

More detailed description will be given under TOLUENE AND DERIVATIVES
Refs: 1) Clift & Fedoroff, Vol **1**, Lefax, Inc (1942), Chap 5, pp 5 & 19 2) Urbański **1** (1964), pp 41, 132, 208

Gamsits. Swiss nonpermissible expls, described in Vol **3** of Encycl, p C443-L, under COMMERCIAL OR INDUSTRIAL EXPLOSIVE. One of them known as **Telsit-Gamsit** contd AN 60, NG 21, NGc 5, Collod Cotton 1.6, woodmeal 0.4 & liq DNT 12% (Vol **3**, p C444-L)

Gap. Maximum distance at which a cartridge (stick) of an expl (such as Dynamite) would explode or detonate by influence (sympathetic deton) of another cartridge of the same or of different expl
Ref: Encycl of Expls, Vol **1** (1960), p XIV (Gap Test)

Gap Tests. Various tests are listed in Vol **4** of Encycl, pp D303-R & D304-L and also under DETONATION (AND EXPLOSION) BY INFLUENCE OR SYMPATHETIC DETONATION, where the following tests are described:
Air-Gap Test (p D397-R);
Booster-Gap Explosive Sensitivity Test of Cole & Edwards (p D398-L);
Card-Gap Sensitivity Test of Cook et al (p D398-R);
Card Test (p D399-L);
Four-Cartridge Test (p D399-L);
Shock-Pass-Heat-Filter (SPHF) Sensitivity Test (p D399-L & D399-R);
Three-Legged Table Sensitivity Test (p D399-R);
Whole-Cartridge Sensitivity Test (p D399-R)
and the following gap tests are listed giving their location in Vols **1** and **3** of Encycl:
Booster Sensitivity Test (p D398-R);
Coefficient de self-excitation (p D399-L);
Halved Cartridge Gap Method (p D399-L);
Wax Gap Test (p D399-R)
Note: "Improved Gap Sensitivity Test for Permissible Explosives" was reported by

R.L. Grant et al of USBurMines at the 11th International Conference of Directors of Safety in Mines Res, Warsaw, Poland, Oct 16–24, 1961 (Two tests are described) (See also R.L. Grant et al, USBurMines, RepInvest No **6947**, 17pp (1967) & CA **67**, 55802b(1967) (Two new gap-sensitivity methods for expls)

Gap Test of Spencer Chemical Co, Kansas City, Missouri, was described as *Wax-Gap Test* in Vol **1** of Encycl, p A354, Note c, under AMMONIUM NITRATE BLASTING EXPLOSIVES

Following are some properties of Spencer Chemical Co AN prill – expls obtd thru the courtesy of S.J. Porter in 1960:

Table G1

Substance	Density g/cc	Deton Vel m/sec	Wax Gap inches
Spencer Prill with 6% oil	0.90	2000	0
Ditto, coated with 3% diatomaceous earth	0.87	2260	2
Ditto, coated with 0.5% diatomaceous earth	0.86	2110	2
Prills and oil mixts *mixts made from* prills mixed with 1.5% diatom earth	0.92	2420	3

Gap Transmission of Detonation Through Various Gases was discussed By T. Kikita & K. Yoneda in KôgyôKayakuKyôkaishi (Journal of the Industrial Explosives Society of Japan) **21**, 21–31 (1960) & CA **55**, 9878(1961)

GAR. Abbr for Guided Aircraft Rocket

Garand, John C (1888–1974). Inventor of the .30-caliber M1 rifle used in WWII. He began working on it at Springfield Armory in the early 1920's and in 1936 it was accepted by the Army, while the Marine Corps accepted it in 1940. Garand's other contributions included numerous tools and gauges, as well as two machine rifles and three semiautomatic rifles

Ref: Anon, Obituary in Miami Herald, Sun, Feb 17, 1974, p 6-B

Garand Rifle. Popular name for the US rifle, caliber .30 M1 (After its designer, John C. Garand, formerly of Springfield Armory). It is replaced now by the M16

Refs: 1) G.M. Barnes, "Weapons of WWII", Van Nostrand, NY (1947), 16–21 2) Glossary of Ordn (1959), 133-R

Garcia (Explosivo). A Spanish blasting expl suitable for use in mines or quarries: K perchlorate 65, Na nitrate 5, Mn dioxide 1.3, sucrose 26, urotropine [hexamethylenetetramine, $(CH_2)_6N_4$] 1.4, iron filings 1.0 & sulfur 1.3%

Note: Total of 101% is as given in the patent

Ref: A.H. Garcia, USP 2215608(1940) & CA **35**, 898(1941)

Gargouse (French). A propellant bag as used in separate-loading ammunition

Garnier, Maurice (1878–1957). French Naval Engineer General (Ingénieur-Générale de l'Artillerie Navale), specializing in ballistics. Numerous publications, mostly in MAF (Mémorial de l'Artillerie Française)

Ref: Anon, MAF **32**, III to XXI (1958)

Garside, Buckley & Yates patented in 1860, 1861 & 1862, complex compns listed in Daniel (1902), p 369 as **Harrison (Poudres).** They were mixts of nitrates (or chlorates) with sugar, lycopodium, starch, charcoal, sulfur & K ferrocyanide

Gas and Gas Laws. Gas is a vaporous or air-like state of matter, defined as a non-elastic fluid in which the molecules are in free movement, and their mean positions far apart. Gases are characterized by their tendency to expand indefinitely, to diffuse and mix readily with other gases, to have definite relations of volume, temperature and pressure and to condense or liquefy at low temperatures or high pressures. One gram-molecule of any gas contains under standard conditions $(6.062\pm0.006) \times 10^{23}$ molecules. This is known as *Avogadro Number N*, because it was defined by *Amadeo Avogadro* (1776–1856), an Italian chemist and physicist who formulated the gas laws (Ref 1, p 89-R)

A "perfect" gas is one which closely conforms to the simple "gas laws" of expansion and contraction, such as **Boyle's Law,** formulated in England by *The Hon Robert Boyle* (1627–1691) (Ref 1, p 141-R) and called in France **Mariotte's Law,** because it was formulated independently from Boyle by *Edme Mariotte* (1629–1684) (Ref 1, p 515-L). This law, called in Germany and Russia Boyle-Mariotte's Law, states that the product of volume and pressure is constant, $pv=k$, at const temp. Another "gas law" states that the vol of a gas at 0°C increases with each °C by 1/273, provided the pressure is constant, and that the pressure increases with each °C, provided the volume remains constant. This law is known as **Charles' Law,** because it was formulated by French chemist *Jacques Charles* (1746–1822) (Ref 1, p 186-R). The so-called **Dalton's Law,** formulated by *John Dalton* (1766–1844), Engl chemist & physicist, founder of atomic theory, states that the pressure of a gas mixture equals the sum of the partial pressures of the constituent gases (Ref 1, p 252-R). The so-called **Gay-Lussac's Law** states that when gases interact, the volumes of the reacting gases and the volume of the reaction product are in simple proportions and can be expressed by whole numbers. This law was formulated by a French chemist and physicist *Gay-Lussac* (qv)

Refs: 1) Hackh's Dict (1944), pp 89-R, 141-R, 186-R, 252-R, 368-R, 371-R & 515-L
2) CondChemDict (1961), 527-L & R
3) Kirk & Othmer (1970), pp 199–205

Gas Analysis or Gasometric Analysis. The methods of gas analysis may be subdivided into the following categories: 1) Volumetric and manometric methods; 2) Specific determinations; 3) Non-specific instrumental methods; and 4) Specific or semispecific instrumental methods (Ref 17)

Among the apparati used in gas analysis, the following are most common: Allen, Burrell, Haldane, Orsat and Petersen

For description of various methods, see

Refs: 1) L.M. Dennis & M.L. Nichols, "Gas Analysis", Macmillan, London (1929) 2) H.A. Daynes, "Gas Analysis by Measurement of Thermal Conductivity", Cambridge Univ Press, London (1933) 3) G. Lunge & H.V. Ambler, "Technical Gas Analysis", VanNostrand, NY (1934) 4) F. Bayer, "Gasanalyse", Enke, Stuttgart (1941) (Reproduced by Edwards Bros, Ann Arbor, Mich) 5) Hackh's Dict (1944), 369 (Table giving composition and properties of industrial gases) 6) V.J. Altieri, "Gas Analysis", Amer Gas Assoc, NY (1945) 7) C.E. Ramslay, Analyst 72, 504(1947) (Volumetric microanalysis) 8) C.T. Langford et al, USP 2429555 & CA 42, 6(1948) (Gas Analyzer Suitable to Determine the Presence of Explosive Gases in Air) 8a) S.H. Ash & E.W. Felegy, USBurMinesBull **471**, 202pp (1948) (Analysis of complex mixts of gases usually encountered in coal mining, such as CO, CH_4, H, $C_2H_4CO_2$, N and air) 9) F.A. Patty, Edit, "Industrial Hygiene and Toxicology", Interscience, NY, Vols 1 & 2 (1948–1949) (Chapter on Atmospheric Analysis) 10) A.W. Gayger & H.T. Darby, AnalChem **21**, 227(1949) 11) H. Levin, AnalChem **21**, 249(1949) 12) F.K. Brooks et al, AnalChem **21**, 1105(1949) (Orsat-type gas analyzer) 13) U.B. Kryukov, V.V. Kamzolkin & A.N. Bashkirov, IzvestiyaAkademii-Nauk, OtdeleniyeTekhnicheskikhNauk **1949**,

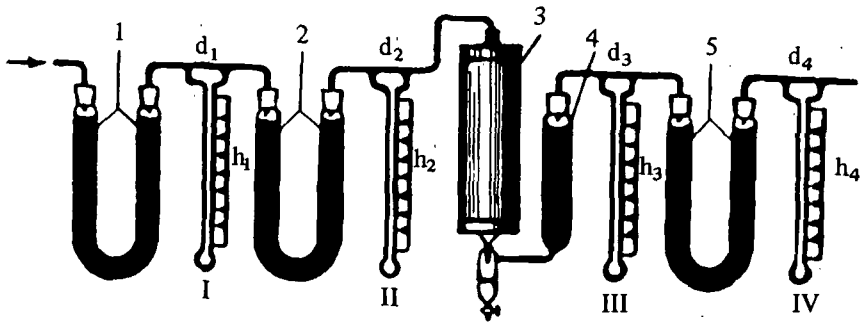


FIG G8

1649–1659 [Detailed description of a new type of gas analyzer suitable for 2, 3, and 4-component mixtures, such as CO_2, CO, H_2 & N_2 (or CH_4). Analysis is conducted automatically at any rate of flow gases. The apparatus consists of the arrangement shown in Fig 8

Gas to be analyzed, contg CO_2, CO, H_2 & $CH_4(N_2)$ passes from a container thru U-tube (1) where moisture is absorbed by Ca chloride and then thru flowmeter (I) (called in Russ "rheometer"), provided with a brass diaphragm (d_1) with diameter of opening ca 0.1–0.5mm. The difference betw the pressure of gas before and after diaphragm is recorded (h_1) in mm of water. When the gas passes thru U-tube (2) filled with Ascarite, the CO_2 is absorbed and the remaining gases pass thru flowmeter (II) with diaphragm (d_2), where the difference of pressure (h_2) is measured. The next step is the passage thru cylinder (3) filled with CuO and heated to 290°C in order to oxidize CO to CO_2 and H_2 to H_2O. The resulting mixture passes thru tube (4) filled with Ca chloride where water is absorbed. Then the remaining mixt goes thru flowmeter (III) with diaphragm (d_3) to U-tube (5) filled with Ascarite to absorb CO_2 formed by oxidation of CO in cylinder (3). The remaining CH_4 (or N_2) goes thru flowmeter (IV) with diaphragm (d_4) and the height (h_4) is registered. Authors give equations allowing one to calculate the amts of components, once h's are known] (Engl translation of this paper is available at the Library of Picatinny Arsenal)

14) L.K. Nash, AnalChem **22**, 108 (1950) (Gas Analysis – in the section "Annual Review of Analytical Chemistry" and in succeeding years) 15) M.P. Matuaszak, "Fischer Gas Analysis Manual for Use with the Apparatus of Orsat Type", Fisher Scientific Co, NY 16) Burrell, "Gas Analysis Apparatus and Burrell Manual for Gas Analysis" 17) R.E. Kirk & D.F. Othmer, Edits, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **7** (1951), pp 73–93: L.K. Nash, "Gas Analysis" (26 refs) 18) W.G. Berl, Edit, "Physical Methods in Chemical Analysis", Academic Press, NY, Vol **2** (1951), pp 387–437: E R. Weaver, "Gas Analysis by Methods Depending on Thermal Conductivity" (91 refs) 19) A. Schmidt, Explosivstoffe **1957**, pp 1–7 (Apparatus for exact gas analysis)

Gas, Black. Same as Carbon Black, briefly described in Vol **2** of Encycl, p C55, under CARBON

Gas, Blast Furnace. It is a weakly combustible gas which passes out at the top of the blast furnace, such as used in recovery of iron from its ores. Compn of purified gas is given by Riegel (Ref), as follows: H_2 1.0, N_2 59.6, CO 26.0, CO_2 13.4. Although its Btu is only 87 per cu foot, it is suitable for several purposes, such as to raise steam for the driving of the blowing engines, as intemal gas fuel for the gas engine driving the air compressor, to heat the stoves so that by means of the latter the incoming blast may be heated. If there is a surplus at the plant, the gas is piped to households of the employees

Ref: Riegel, "Industrial Chemistry" (1942), pp 780–81

Gas, Blau. A fuel gas for airships obtd by cracking *gas oil* at 550–600°. It is a mixture of propane, butane, pentane and hexane contg some hydrogen & methane in soln under pressure. The soln is handled in strong steel cylinders

Refs: 1) Hackh's Dict (1944), 133-L (Blau Gas) 2) E.R. Riegel, "Industrial Chemistry", Reinhold, NY (1949), 306

Gas Bubbles Effect on Detonation of Explosives. See F.P. Bowden et al, ProcRoySoc **188A**, 291–311 (1947) & CA **41**, 3627 (1947) (The detonation of liq expls by gentle impact; the effect of minute gas spaces)

Gas Cannon Device, patented by Peake in 1934 (Ref), was claimed to be an improvement for previously proposed "gas cannons". The object of this device was to provide a gas generating propellant in measured form. It consisted of pellets sol in water, coated with material, which although not readily

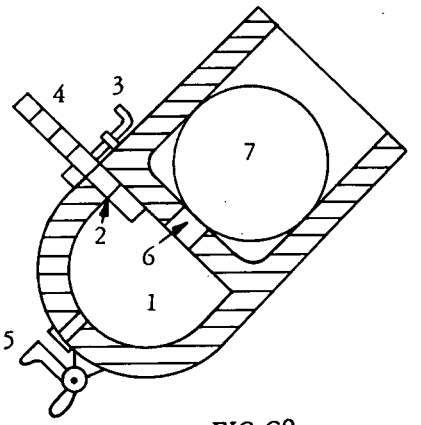


FIG G9

attacked by moisture nevertheless dissolved when placed in a generating chamber in contact with a sufficient amt of water. In one of the varieties, (Fig G9) the material was in the form of carbide stick (4) consisting of several sections. The "inlet port" (2) of the "generating chamber" (1) was closed by means of "cover" (3), which was pivoted and adapted to swing over, or away from the port (2). For loading the chamber (1), the cover (3) was removed and the carbide stick (4) inserted Its sections were broken from the stick by means of a twist or bent and, after placing them in chamber (1), the cover (3) was placed over inlet port (2), so that no gas could escape from (1). When water was introduced in chamber, the protective coating of carbide sections was dissolved and the carbide reacted with water forming acetylene. As soon as all carbide dissolved, the striking mechanism (5) was to act on a primer, in order to explode the mixture of acetylene and air. Then the pressure of exploding gases escaping thru opening (6) propelled ball (7)

Instead of carbide stick, spaced, coated carbide balls could be used

Ref: E.S. Peake, USP 1970017 (1934) & CA **28**, 6314 (1934)

Gas Cannons. The first cannons were small, crudely made cast metal pots shaped like a vase which fired iron darts. The invention of gun powder about the middle of the 13th century made possible the use of cannon to throw projectiles by use of expanding gas. In the first cannons, the propellant was ignited by thrusting red hot iron bars or spikes in the charge thru an aperture. Those that more nearly resemble the cannon of a later period were made of wooden staves fitted together, barrel-like and held tight with many folds of wet rawhide. Later when the use of iron became known the same construction was followed, square bars of iron being placed lengthwise and held together with iron hoops put on hot

Refs: 1) J.R Newman, "The Tools of War", Doubleday Doran & Co, Inc, NY (1942), pp 69–75 2) Anon, "Field Artillery, Basic", Military Service Publishing Co, Harrisburg, Pa (1943), pp 185–89

Gas, Carbon Disulfite. A flammable toxic gas described in Vol **2** of Encycl, p C60-R & C61-L & R

Gas, Carbon Monoxide. A combustible gas described in Vol 2 of Encycl, p C62-L

Gas, Carburetted Water. See under Gas, Water

Gas, Chemical Warfare (CWG). See Vol 2 of Encycl, under CHEMICAL AGENTS OR CHEMICAL WARFARE AGENTS, pp C165-R to C171-R

Gas Chromatography and Gas-Liquid Chromatography. See Vol 3 of Encycl, p C293-R, under CHROMATOGRAPHY

Gas Cleaning and Purification. *Gas cleaning* means the removal of impurities existing in the form of suspended liquid or solid particles, such as vapors, mist, fog, smog, smoke or dust. The impurities might also include gaseous substances that are objectionable or obnoxious, and their removal is usually called *purification*

Gas cleaning may be accomplished either by *mechanical methods* (such as gravitational settling, centrifugal separation, filtration, ultrasonic precipitators, spray towers, mechanical scrubbers, etc) or by *electrical methods* (such as electrostatic precipitation by Cottrell Method, etc)

Purification consists of removal of gaseous impurities by means of absorption or adsorption processes (such as by activated charcoal)
Refs: 1) F. Fraas & O.C. Ralston, IEC **32**, 600–04(1940) (Electrostatic sepn of solids from gases) 2) H.F. Johnstone & M.H. Roberts, IEC **41**, 2417(1949) (Deposition of aerosol particles from moving gas streams) 3) H.W. StClair, IEC **41**, 2434(1949) (Agglomerates of smoke, fog or dust by sonic waves) 4) H.W. Danser & E.P. Neumann, IEC **41**, 2439(1949) (Industrial sonic agglomeration and collection systems) 5) R.M. Reed & N.C. Updegroff, IEC **42**, 2269(1950)(Removal of hydrogen sulfide from industrial gases) 6) J.H. Perry, Edit, "Chemical Engineers' Handbook", McGraw-Hill, NY (1963), pp **20**-(62–96) 7) R.E. Kirk & D.F. Othmer, Edits "Encyclopedia of Chemical Technology", Interscience, NY, Vol **10** (1966), pp 329–352: S. Sylvan & R.F. Logsdon, "Gas Cleaning" (12 refs)

Gas and Coal Dust Explosion. Title of a series of papers published by K. Matsumoto in JMiningInstJapan **68**, 260–5 & 379–82 (1952) & CA **48**, 3691(1954). The papers include adsorption of mine gases by the coal dusts; heat of adsorption of mine gases by coal dusts and ignition temperature of the coal dusts

Gas, Coal. Accdg to Riegel (Ref), coal gas is a combustible gas obtd by distilling bituminous coal in small horizontal and vertical retorts. The primary object of distn of coal is gas. As coal gas is high in illuminants (benzene and ethylene), it can serve as *illuminating gas*
(See also under "Coal Processing for Obtaining More Valuable Products", in Vol 3 of Encycl, p C379-L)
Ref: E.R. Riegel, "Industrial Chemistry", Reinhold, NY (1942), p 153–54 (Retort for distillation of coal); pp 270–71 (Combustible and illuminating gas)

Gas, Coal Mine or Firedamp. A mixture of methane (CH_4) and air, usually present in "gaseous (fiery) coal mines", such as soft (bituminous) coal mines

Explns of firedamp and its mixtures with coal dust are described in Vol 3 of Encycl, under "Coal Mine Explosions and Fires", pp C360-R to C367-R

Gas, Coke-Oven. It is a combustible gas obtd, accdg to Riegel (Ref), by distilling bituminous coal in larger retorts than those used in manuf of *coal gas* (See Gas, Coal). Both coke and gas are primary objects of manuf. Following typical compn of "straight coke-oven gas" is given by Riegel: CO_2 1.8, C_6H_6 0.9, C_2H_4 2.3, O_2 0.8, CO 5.5, H_2 55.5, CH_4 30.3, N_2 9.2; total combustibles 88.2 and Btu per cu foot 555
Ref: E.R. Riegel, "Industrial Chemistry", Reinhold, NY (1942), pp 254–55 (Coke ovens); pp 270–71 (Coke-oven gas)

Gas, Combustible. Any gaseous substance or mixture consisting principally of carbon and hydrogen contg gases used for illuminating or heating purposes may be called combustible gas. These include blast furnace gas, carbon monoxide gas, coal gas, coke-oven gas, fuel gas, natural gas, oil gas, producer gas, tar gas, water gas and wood gas
Refs: 1) J.J. Morgan, "Gas Engineers' Handbook", AmGasAssoc, NY (1934) 2) H. Hollings, "Gas Manufacture", Benn, London, v 1(1934) 3) P. Dolch, "Wassergas", Barth, Leipzig (1936) 4) "Handbuch der Gas Industrie Generatoren", Oldenburg, München (1940) 4a) E.R. Riegel, "Industrial Chemistry", Reinhold, NY (1942), 270–77 5) National Research Council, "Chemistry of Coal Utilization", J. Wiley, NY (1945) 6) Batelle Memorial Inst, "Economics of Fuel Gas from Coal", McGraw-Hill, NY (1950) 7) A.J. Johnson & G.H. Auth, "Fuels and Combustion Handbook", McGraw-Hill, NY (1951) 8) R.E. Kirk & D.F. Othmer, "Encyclopedia of Chemical Technology", Interscience, NY, v 10(1966), pp 353–442: M.A. Elliott & H.R. Linlen, "Manufactured Gas" (309 refs)

Gas Conditioning. Accdg to Riegel (Ref), gas conditioning means removing from a gas (usually air) suspended non-gaseous particles or droplets, as well as foreign gases and vapors, and adjusting its water-vapor content and temperature to predetermined levels
Ref: E.R. Riegel, "Chemical Machinery"; "An Elementary Treatise on Equipment for the Process Industries", Reinhold, NY (1944), pp 460–72 (Detailed description with numerous refs of air-conditioning and of some industrial gas conditioning)

Gas Dispersion. The dispersion of gas as bubbles in a liquid or in a solid (such as rubber, soap, plastic) is effected for one of the following purposes:
1) *Agitation of liquid phases.* It is produced by blowing the air or other gas (from a sparger etc) to the lower part of the container of liquid. This is usually done in cases when the containers are of such size or of such unsymmetrical shape as to make mechanical agitation ineffective or too expensive. Another case is when mechanical agitation is considered unsafe, as in the preparation of some expls. For instance, in the nitration of glycerin, air agitation is still used in some European countries
2) *Foam or froth production.* This is done for other purposes than agitation. For instance, in the case of the flotation method of ore concentration, frothing causes the mineral particles of ore to float on the surface of the water. In some models of fire extinguishers, foam is produced by the action of carbon dioxide (evolved by the action of sulfuric acid on soda ash) in order to increase the volume of water in the extinguisher and to introduce bubbles of gas, which does not support combustion. In the case of solids such as soap, foam is produced to make the soap lighter so that it can float on the surface of water. In the case of plastics, air is introduced to decrease the specific gravity of the material; in the case of ice cream, to get more product per unit weight
3) *Gas-liquid contacting to promote absorption, or stripping.* This is usually accomplished by bubbling the gas thru a liquid in the same manner as for agitation
Refs: 1) S. Berkman & G. Egloff, "Emulsions and Foams", Reinhold, NY (1941) 2) J.H.

Perry, Edit, "Chemical Engineers' Handbook' McGraw-Hill, NY (1950), pp 1175–79: S.A. Miller, "Gas Dispersion"

Gasdruckpatronenen. See Gas Pressure Cartridges (Ger)

Gas Engines or Internal Combustion Engines. A prime mover, the fuel of which is burned within the engine. Examples are gasoline piston engines in passenger automobiles, outboard engines for motor boats, small units for lawn mowers, also diesel engines for trucks, tractors etc. Characteristic features common to all commercially successful internal combustion engines include 1) compression of air; 2) the raising of air temperature by the combustion of fuel in this air at its elevated pressure; 3) extraction of work from the heated air by expansion to the initial pressure and 4) exhaust

Ref: D.N. Lapedes, Edit, "Mc-Graw-Hill Encyclopedia of Science & Technology", Vol **7**, McGraw-Hill, NY (1971), pp 226–39

Gaseous Discharge Lamps. Gaseous discharge lamps consist of an electrically operated source of radiant energy characterized by the emission of radiation from a stream of ionized gas carrying current between electrodes in the lamp (See Fig G10). Lamps in common use include fluorescent, mercury-vapor and neon lamps. In general, gaseous discharge lamps provide their characteristic energy at higher efficiencies than other sources. All types of gaseous discharge lamps possess a negative resistance characteristic; that is, the resistance in the lamp envelope decreases with an increase in current. To prevent lamp failure from rapid rise of current, a current limiting element, or ballast, is used usually external to gas discharge envelope

Refs: 1) W.E. Forsyth & E.G. Adams, "Fluorescent and Other Gaseous Discharge Lamps", Murray Hill Books, NY (1948)

2) C. Zwikker, Edit, "Fluorescent Lighting", Elsevier Press Inc, NY (1952) 3) D.N. Lapedes, Edit, "McGraw-Hill Encyclopedia of Science & Technology", Vol **14**, McGraw-Hill Inc, NY (1971), p 305

Gaseous Detonations and Explosions. See in Vol **4** of Encycl: "Detonation (and Explosion) in Gases", pp D351–360; also "Detonation (and Deflagration) in Gases; Determination of Ignition Points", p D360 and "Detonation (and Explosion) of Gases, Vapors and Dusts"; "Development (Transition) from Burning (Combustion) or Deflagration", pp D360-R to D363-R

Addnl Refs: A) S.G. Lipsett, CanadChemProcessInds **30**(3), pp 41–6 & 48(1946); CA **40**, 3265(1946) (Gaseous and dust explns are discussed from the point of view of safety in the home and in the factory)

B) Ya.B. Zel'dovich & N.N. Simonov, ZhurFizKhim **23**, 1361–74(1949); CA **44**, 2753

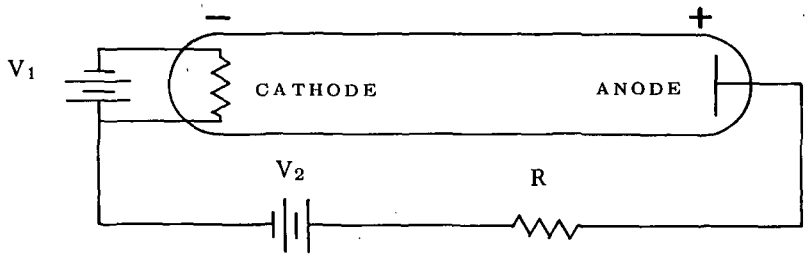


FIG G10 Gaseous discharge tube in series with a DC voltage V_2 and a resistor R. The voltage V_1 serves to heat the cathode.

(1950) (Theory of the spark ignition of expl gas mixtures)
C) K. Matsumoto, JMiningInstJapan **70**, 463–66 (1954); CA **49**, 6607 (1955) (Gas and coal dust explns and adsorption of the mine gas by Japan coals Joban & Ube)
D) R.G.W. Norrish et al, "Kinetic Studies of Gaseous Explosions", 5thSympCombstn, Reinhold, NY (1955), pp 651–56 (5 refs)
E) G.G. Bach et al, "Initiation Criteria for Diverging Gaseous Detonations", 13thSymp-Combstn, The Combustion Institute, Pittsburgh, Pa (1971), pp 1097–1180

Gaseous Metal Treatment. In order to protect ordinary iron and steel items from rusting, they were coated, in Germany, by the diffusion of chromous chloride vapor at high temperature. The method was briefly described in Ref 1 and listed in Ref 2
Refs: 1) BIOS Final Repts **839**(1946) & **1534**(1946) 2) PATR **2510**(1958), p Ger 63-R

Gaseous Mines or Fiery Mines. Under this term are known coal mines contg a mixture of firedamp (impure methane) and air in such proportions that a flame or spark would result in spontaneous expln. Mixts contg 5 to 12% of firedamp are usually considered expl, and the presence of coal dust in such mixts makes them more dangerous. The most dangerous is the mixt contg 9.5% methane. Only "permissible expls" are allowed to be used in gaseous mines
Ref: PATR **2700**, Vol 3 (1966), pp C361 & C369

Gases, Detonation (and Deflagration) in. See Vol 4 of Encycl, p D360

Gases, Detonation (and Explosion) in. See Vol 4 of Encycl, pp D351 to D360

Gases, Dusts and Vapors, Detonation (and Explosion) of. See Detonation (and Explosion) of Gases, Vapors and Dusts in Vol 4 of Encycl, pp D360-R to D363-R

Gases, Fuel. Any combustible gas that furnishes heat on combustion, such as coal gas, natural gas or water gas
Ref: Hackh's Dict (1944), pp 359-L & 368-R

Gases Liberated from Explosions Initiated by Impact. It has been known for a long time that in a true deton of HE, the major products are CO_2, CO, H_2O, N and H, while in thermal decomposition of expls, more complicated mixtures of NO_2, NO, N_2O, CO_2, CO, H_2, aldehydes etc, result. As it is of interest to know what kind of products are obtained when an expl is initiated by impact, Robertson and Yoffe (Ref 8) tested NG, PETN and RDX in a special vacuum impact machine. Their results showed that gases developed on impact approximate more closely those developed on thermal decomposition than those on deton. The results are in harmony with the hypothesis of a thermal origin of impact-initiated explns and with the slow initial burning velocities observed with the rotating drum camera
Refs: 1) R. Robertson, JCS **95**, 1241 (1909) 2) R. Robertson & W.E. Garner, ProcRoySoc **A103**, 539 (1923) 3) A. Haid & A. Schmidt, SS **26**, 253, 293 (1931) 4) A. Appin, J. Chariton & O. Todes, ActaPhisicochim-(Russia) **5**, 655 (1936) 5) J. Taylor & C.R.L. Hall, JPhysCollChem **51**, 593 (1947) 6) F.P. Bowden & O.A. Gurton, Nature **161**, 148 (1948) 7) A. Yoffe, Nature **161**, 349 (1948) 8) A.J.B. Robertson & A. Yoffe, Nature **161**, 806–07 (1948) 9) P. Gray, TransFaradaySoc **46**, 848–52 (1950) & CA **45**, 2669g (1951)

Gases Produced on Decomposition of Smokeless Propellants. Their volume was detd by P. Tavernier, MP **35**, 259–72 (1953)

Gases Produced on Deflagration of Smokeless Propellants. Their Nonideal Behavior was discussed by P. Tavernier, MP **37**, 269–304 (1955) & CA **51**, 1610(1957)

Gases Produced on Explosion or Detonation of Explosives. The usual gases are CO_2, CO, H_2O, N_2, O_2, NO_2 and H_2. For determining the compn of gases evolved on expln or deton, the test is conducted in a closed vessel, such as Bichel Bomb (described in Ref 1, pp 91–95 and in Vol 3 of Encycl, pp C331-R & C332-L), by the procedure described here under "Gas Volumes Produced on Explosion or Detonation of Explosives". After cooling the bomb, the gases are collected in a gasometer, their volume calcd to 0° & 760mm Hg and then they are analyzed by one of the methods listed here under "Gas Analysis"

In ADL Report (Ref 3) are given compns of gases developed on deton of TeNMe (Tetranitromethane) alone and its mixture with an amt of RDX to obtain an Oxygen Balance to CO_2 equal to +10%. See Table G3

Table G3

Explosives	TeNMe (alone)	TeNMe (+RDX)
OB to CO_2	+49%	+10%
Gases, Volume Percent		
CO_2	38.4	40.1
O_2	8.2	–
H_2	–	3.0
CO	0.6	5.7
N_2	45.5	51.2
NO_2	7.3	–
Total Explosive accounted for	100%	100%

Composition of gases produced on deton of the following expls and given in Stettbacher (Ref 2, p 75) are reproduced here in Table G2

TABLE G2

For 1 kg of Explosive	Blasting Gelatin 92/8	PETN	NC 13.3%N	Tetryl		Picric Acid		TNT
Density	1.45	1.54	1.3	0.9	1.56	1.00	1.45	1.52
Gases:								
CO_2	13.27	9.34	8.05	3.02	5.59	4.49	7.88	5.3
CO	0.83	0.44	10.29	18.83	10.85	17.6	10.18	8.79
C (Soot)	–	–	2.5	0.3	5.8	0.8	0.8	15
H_2O	11.17	11.05	9.93	3.03	5.91	4.02	4.60	7.05
H_2	0.17	1.53	2.45	3.64	1.89	1.70	1.08	1.69
O_2	0.30	0.40	0.14	–	–	–	–	–
NO	0.50	1.0	–	–	–	–	–	–
N_2	6.15	5.82	4.81	7.73	7.82	5.02	5.81	5.2
NH_3	–	–	–	0.3	–	0.1	0.3	0.9
CH_4	0.03	–	0.15	0.48	0.27	0.16	0.12	0.03
C_nH_n	–	–	–	0.02	0.03	0.04	0.01	0.05
HCN	–	–	–	1.2	0.6	0.66	0.33	1.4
$(CN)_2$	–	–	–	0.2	0.58	1.2	0.4	0.1
CO_2/CO	16	1.5	0.8	0.16	0.5	0.25	0.7	0.6
Gas Volume, liters/kg	727	798	800	860	750	780	698	688

Experimental procedures for determination of gaseous products of expls on exploding, by means of Bichel Pressure Gage and Crawshaw-Jones Apparatus are described in Ref 1, but no compns of gases are given

Refs: 1) Ch.E. Munroe & J.E. Tiffany, "Physical Testing of Explosives", USBur-Mines Bull **346**(1931), 91–99 2) A. Stettbacher, "Schiess- und Sprengstoffe", Barth, Leipzig (1933), p 75 3) ADL, Synthesis HE's, 2nd Report (1951), 330–335 4) J. Sinabell, Explosivst **8**, 141–7(1960) & CA **55**, 3981(1961) 5) J. Tranchant & M. Moreau, MP **42**, 445–56(1960) & CA **55**, 12149(1961) 6) H. Schubert & F. Volk, Explosivst **14**(1), 1–8(1966) & CA **64**, 15667(1966) 7) D.L. Ornellas, USAtEnergyComm **UCRL-70444**, 7pp (1967) & CA **68**, 41753(1968) 8) D.L. Ornellas, JPhysChem **1968**, 72(7), 2390–5 & CA **69**, 53315(1968)

Gas-Generating Cartridges. See Vol **2** of Encycl, under Cartridges and Cartridge-Actuated Devices (CAD's), called Propellant Actuated Devices (PAD's) by US Ordnance Corps and Power Cartridges by USNaval-Ordnance, pp C70-R to C72-R

Gas-Generating Nondetonating Compositions are described in the following refs:

Refs: 1) J. Taylor, USP 2159234 & CA **33**, 7116(1939) (Gas-generating nondetonating compn suitable for use in blasting. It consists of a pressure-resistant, pressure-responsive container, a deflagrating ignition means and a deflagrating gas-generating compn capable of undergoing self-sustaining decompn without deton) 2) J. Taylor & A.C. Hutchison, USP 2604391(1952) & CA **47**, 1932(1953) [High yield of gases and at a greater rate is produced on thermal decompn of NGu or GuN (Guanidine Nitrate) if a small amt (2 to 20%) of Cu powder or Cu compd (such as CuCl or $Cu_2C_2O_4$) is incorporated. These substances act as sensitizers. The addn of 1% V_2O_5 increases further the rate of burning] 3) Ibid, USP 2653086(1953) & CA **48**, 2376 (1954) [Two mixtures of gas generating, non-detonating compns are described: a) K chromate 20 & NGu (Nitroguanidine) 80% and b) NGu 80, Amm dichromate 10, DPhDEtUrea 5 & charcoal 5%, mixed in a ball mill and pressed in cylindrical form. The rate of burning of pressed chge was 0.07 inches/sec at a pressure of ca 100 atm] 4) A.C. Hutchison, USP 2710793(1955) & CA **49**, 13652(1955) (Solid gas-generating units) prepd from 89–98.8% GuN or Nitroguanidine, 0.2–10% molybdic, ceric, or vanadic oxide and 0.25–1% asbestos fiber 5) J.F. Williamson, BritP 724060(1955) & CA **49**, 14325(1955) (Nondetonating, gas-generating expl charge for use in fiery mines prepd from $Mg(NO_3)_2.6H_2O$, AN & wood flour; packed in a ported venting head container 6) A.C. Hutchison, USP 2744816(1956) & CA **50**, 12444–46(1956) (Solid gas-generating charges exemplified by GuN 88.75, 2,4-dinitroresorcinol 10, V_2O_5 0.5, asbestos fibers 0.5 and KNO_3 0.25 (nitro or nitroso phenols and V_2O_5 are part of claim) 7) M. Boyer, USP 2981616 (1961) & CA **55**, 25260(1961)(Gas-generating compns for pressurizing rocket-propellant tanks consisting of mixt of an azide and an oxidizing compd, eg, NaN_3 60 & Na_2O_2 40%) 8) J.W. Rabern & N.J. Wilkaitis, GerP 1109577 (1958) & CA **56**, 6232(1962) (Gas generator based on AN and a binder) 9) A.T. Camp & F.G. Crescenzo, USP 3102834(1963) & CA **59**, 11182(1963) (Gas generator based on NC 48, NG 31.2, triacetin 16.25, 2-nitrodiphenylamine 1, Ethyl Centralite 1, PbO 1, CuO 1, cellulose acetate 0.5 and candelilla wax 0.05% has a burning rate of 0.225 inches/sec)

Gas Hydrates. They are gases (such as ethylene, propane and isobutane) which form *clathrate* (See Ref 2) compounds on contact with water. These compds are solid and insol in w. They usually form and exist at relatively low temps and high pressures. Anywhere from 6 to 18 moles of water may combine with each mole of gas, depending upon the nature of the gas

Interest in the gas hydrates for many years was generated mainly because of the nuisance of such compds forming in gas pipelines. In recent years, the compds have been proposed as a means of precipitating water from salt solns (or sea water), thus yielding potable water
Refs: 1) CondChemDict (1961), 527-R & 528-L 2) PATR **2700**, Vol **3**(1966), p C327 (Clathrates and Other Inclusion Compounds)

Gas Ignition Point. Discussion of relationships between ignition point (autoignition) and drop ignition point (spontaneous ignition). Derivation of a formula for calcg drop point temps
Ref: E. Oehley, ChemIngTech **26**, 97–100 (1954) & CA **48**, 5501 (1954)

Gas, Illuminating. A gas used for production of light. It is prepd by distillation of bituminous coal in small horizontal or vertical retorts. Other combustible gases may be used for this purpose, provided they contain sufficient amts of illuminants, such as benzene or ethylene. Some illuminating gases contain "natural gas" mixed with other gases (See also under Gas, Coal)
Ref: Riegel, "Industrial Chemistry" (1942), 270–71

Gasket Materials Used in Ordnance. Various types are described in
Refs: 1) ORDP **20-307**, "Ordnance Materials Handbook", (Gasket materials, nonmetallic) (1956) 2) J.H. Perry, Ed, "Chemical Engineers' Handbook", McGraw-Hill, NY (1963), Table 23-11 (Important Properties of Gasket Materials) 3) Kirk & Othmer, Vol **14**, pp 443–61 (1967), E.W. Fisher: "Packing Materials"

Gas, Lachrymatory. See Gas, Tear

Gas, Laughing. Same as Nitrous Oxide, N_2O, used as a dental and surgical anesthetic and to preserve perishable foods
Ref: Hackh's Dict (1944), 368-R & 580-R

Gasless Delay Detonators, Electric (German). These detonators used during WWII were prepd as follows:
a) Detonator shells (Hülse) of Al having an outside diam of 7.20mm and lengths ranging from 52.5 to 85mm (depending on the delay required) were thoroughly cleaned and dried before loading. Cu shells also were used
b) Tetryl was loaded in two increments (a total 0.7g) to serve as a base chge. This was followed by priming charge of 0.3g of 60/40–LA/LSt mixture and a perforated reinforcing cup, all pressed at 250kg/sq cm
Note 1: Tetryl, LA and LSt were previously dried to a max moisture content of 0.1%
c) After keeping the loaded detonators for 3 days at 50°C in order to remove all traces of moisture, 50ml of loose intermediate compn was placed on top of the reinforcing cup
Note 2: The intermediate compn (powdered mixt of Sb and $KMnO_4$) formed a loose connection between the delay compn (to be loaded next) and the priming compn, LA/LSt. The intermediate compn burned with a strong flame which facilitated the ignition of the LA/LSt mixt. Misfires could take place if the delay mixt were placed in direct contact with LA/LSt
d) The next step was to press on top of the intermediate mixt the delay element contg a compressed powdered mixt of Sb & $KMnO_4$. The detonator shell was then crimped just above the upper end of the delay sleeve in order to provide a seal for the "Mipolam" sealing plug, described in Ref 4, p Ger 113-R
Note 3: Accdg to Ref 1, pp 5–6, the gasless delay powder (also called "gasless delay fuze powder") consisted of about 70% Sb pdr & 30% $KMnO_4$ pdr for slow burning, or about 46% Sb & 54% $KMnO_4$ for fast burning. The permanganate was ground in a disc or plate crusher mill to approx 80 mesh. The antimony

was ground from lumps in a vibratory ball mill and the pdr was transferred by a screw feed into an air separator. The fines which did not exceed 10 microns in size were collected and blended with the permanganate by means of a tumbling mill. The resulting mixt was compressed into tablets in a rotary multiple punch press. The tablets were then broken ina plate crusher mill and the resulting powder was used loose as an intermediate chge and compressed to form a delay element e) The fusehead assembly (qv) consisting of bridge wire, igniter bead, two lead-in wires (insulated by Mipolam), and the Mipolam plug, was inserted in the detonator shell in such a manner that the plug rested on the shoulder of the detonator shell formed by crimping. A second crimping was then made above the plug and the lead-in wires were connected to a source of electricity when the detonator wss to be fired

Refs: 1) CIOS Report **24-3**(1945), pp 5-6
2) BIOS Final Rept **833**(1946), Item 2, Appendix A3 3) "Manufacture of German Detonators and Detonating Compositions", PB Rept 95613(1947), Sectns B to L, incl
4) PATR **2510** (1958), pp Ger 63 & Ger 66

Gasless Delay Elements and Detonators Employing Them. See Vol **4** of Encycl, Section 4, Part F, pp D863-R to D868-R

Gasless Delay Fuze Primers. See Vol **4**, pp D868-R to D869-L

Gasless Delay Powders for Ammunition Fuze Applicators are described in the following Picatinny Arsenal Technical Reports:

Refs: 1) D. Hart, "Gasless Powders for Delay Elements of Fuzes", PATR **1239**(Feb 1943)
2) D. Hart, "Gasless Powders for Delay Elements of Fuzes", PATR **1281** (April 1943)
3) D. Hart, "Gasless Powders for Delay Elements of Fuzes", PATR **1406** (March 1944)
4) S. Sage, "Manufacture of Barium Chromate Delay Powder on a Semi-Plant Scale", PATR **1432** (July 1944)
5) D. Hart, "Gasless Powders for Delay Elements of Fuzes", PATR **1513** (March 1945)
6) J.E. Osmun, "Surveillance Tests on M16A1 Primer Detonators Containing Type I, Class B Delay Powder", PATR **1546** (July 1945)
7) M.C. Epton, "Long Range Development of Delay Powders for Ammunition Fuze Application (Bomb Fuzes)", PATR **1686** (April 1948)
8) D. Hart, "Long Range Development of Delay Powders for Ammunition Fuze Application," PATR **1733** (June 1949)
9) M.T. Hedges & T.R. Mahler, "Surveillance Study of Nickel-Zirconium Type Delay Powder for M205 Hand Grenade Fuze", PATR **1952** (Aug 1953)
10) T.J. Mahler & M.C. Epton, "Study of Nickel-Zirconium Type Delay Composition for Use in 11- to 14-Second Primer Detonator", PATR **1976** (Oct 1953)
11) P. Rochlin, "Mass Spectrometric Analyses of Gaseous Combustion Products from Delay Powders (Research and Development of Delay Powders for Ammunition Fuze Application-General) ' ', PATR **2006** (March 1954)
12) D.J. Zouder, T.J. Mahler & M.T. Hedges, "Development of Zirconium-Nickel Alloy Delay Powder for M204A1 Hand Grenade Fuzes", PATR **2228** (Jan 1956)
13) B. Werbel, "Development of Delay Powders", PATR **2249** (Sept 1955)
14) B. Werbel & S. Lopatin, "Development of Delay Powders", PATR **2477** (April 1958)

Gasless Explosives Investigated in Russia. The following mixts, which produced no gaseous materials in either the initial or final stage, were investigated by Belyaev (Ref):

Mixture No 1. $3.6KClO_3 + FeSi_{4.65} = 3.6KCl + 0.5Fe_2O_3 + 4.65SiO_2$

Mixture No 2. $KClO_3 + 2Al = KCl + Al_2O_3$

By using No 8 detonator and Lead Block Compression Method, which was a modification of Hess' Method, described in Vol **3** of Encycl, p C492 as the "Brisance Meter of Hess", Belyaev obtd the compression values in mm shown in Table G4

Table G4

	Mixture No 1			Mixture No 2		
Diam of chge, mm	40	58	77.5	40	58	77.5
Wt of chge, g	50	240	480	50	240	480
Compression, mm	0	0.7	15.1	5.4	16.73	21.55

Detn velocities as detd by method of Dautriche, described in Vol 3 of Encycl, p C311-R, were:

Mixture No 1: 1170m/sec in a tube 20mm diam and 1270 in a tube 27mm diam. No densities were indicated

Mixture No 2: 1370m/sec in a tube 21mm diam and 1370 in a tube 40mmdiam. No densities were indicated

Note: Both mixts contained KCl in their products of expln. As the temp of expln was higher than the critical for KCl, it means that KCl should have been in the vapor state during expln process and consequently behaved like a gas. As the volume of KCl vapor was small, in comparison with those given off by ordinary expls, it may be considered negligible and the mixts gasless

Ref: A.F. Belyaev, ComptesRendusAcadScie (Russia) **46**, 107–09(1946) & CA **40**, 4531(1946)

Gasless Ignition Powders: 1) *A-1A Composition:* Zr 65.0±1.0, iron oxide 25.0 1.0 & diatomaceous earth 10.0±1.0%; 2) *F-33B Composition:* Zr 41.0±1.0, iron oxide 49.0 1.0 & diatomaceous earth 10.0±1.0%

Ref: Specification MIL-P-22264 Wep, Amend 2, 1962

Gasless Mixture 668/Mn: Mn 10, Cu 30, Si 30 & PbO_2 30%. Pressure on heating of this mixture was developed by sublimation of metals

Ref: W.A. Show, "Development of an Incendiary Pellet", Tech Command Army Chem Center, Md Rept **TCR-59** (May 1950)

Gasless Powders for Delay Elements of Fuzes. Many compns were developed by Dr Hart in the Pyrotechnic Laboratory of Picatinny Arsenal. They are described in the following Picatinny Arsenal Reports:

1239 (Feb 1943). Compositions contg Ba chromate, manganese and sulfur show promise for use in M54 Time Fuze and in the M16 Delay Element

1281 (April 1943). New igniter compn for ammunition contains Pb chromate 50.0, manganese 32.5 & sulfur 17.5%

1406 (March 1944). New fuze powd contains Ba chromate 74–77, manganese 20–22 & sulfur 3–4% and is suitable for use in 8–11 second M16A1 Delay Element of bomb fuzes

1513 (March 1945). Improved 8–11 second delay powder contains Ba chromate 70.9, manganese 27.1 & sulfur 2.0% with 2–3 parts ethyl cellulose added

Gasless Reactions. Gasless compns consist of finely powdered reducing agents and oxidizing agents mixed together and often consolidated by compression. The chem processes are exothermic oxidation-reduction reactions, commonly involving transfer of oxygen from a metallic oxide or oxy-salt to a metal

Investigations have been carried out on compressed mixts of finely divided iron & Ba peroxide, and iron & K dichromate, which undergo incandescent reaction when ignited. Also systems using finely divided iron, manganese & molybdenum as reducing agents and Ba peroxide, K permanganate and the nitrates of K, Pb, Sr & Ba as oxidizing agents were studied. Three other systems – iron/K dichromate, sulfur/Ba peroxide and Si/K dichromate were investigated in a range of proportions and the heat evolved in the reactions was measured

One of the best-known gasless reactions is Thermite which is comprised of a mixt of Al powder & iron oxide:

$$3Fe_3O_4 + 8Al \rightarrow 4Al_2O_3 + Fe$$

The quantity of heat released is 850cal/g and the temp reached is enough to melt the iron. This reaction and its applications were discovered & developed by Hans Goldschmidt (1861–1923). The Thermite type reaction was used in making incendiary bombs during WWII. Thermite is more effective when used with

addnl Al powder, Ba nitrate & sulfur as an igniting means for Mg bombs

One of the most useful & important applications of gasless compns is in delay elements of electric detonators. There is a vast amt of info in the patent literature describing these compns. One commonly used mixt is antimony & K permanganate:

$2Sb + 10/3KMnO_4 \rightarrow 10/3MnO_2 + Sb_2O_5 \cdot 5/3K_2O$

which liberates 390cal/g. A Canadian Industries Limited patent describes a short period delay detonator comprising a homogeneous mixt of 70/30 Misch metal/Al 13.4, silicon 24.1 & red lead 62.5% yielding gasless reduction products. One ICI patent claims a compn of red lead & powdered Ti or Zr can be used for delays of 10–1700 milliseconds, and is insensitive to ignition by friction

In order to initiate the delay element it is necesssry to use an electric fusehead which is also gasless. One such fusehead is made by dipping the bridge wire in a suspension of a mixt of Zr powder 50 & Pb 2-Mononitroresorcinate 50% in a 5% soln of NC in amyl acetate

Other interesting applications of gasless compns are those used in the hot tube safety igniter for initiating blasting devices of the "Hydrox" type. The heat of reaction of a chge of Zn powder 60 & K permanganate 60% is sufficient to ignite Hydrox powder, but not high enough to ignite methane/air mixts or coal dust/air mixts which occur in fiery coal mines. Also developed during WWII was a self-heating food can capable of heating its foodstuffs content to a desirable temp in a few mins and yet not much bulkier than an ordinary can contg the same amt of foodstuff. The heating mixt most suitable is a 50/50 mixt of finely powdered Ca silicide & Fe_3O_4. Easy ignition of this mixt is ensured if a priming layer of Ca silicide/red lead 30/70 with the addn of 10% china clay to slow the rate of reaction

Ref: J. Taylor, "Solid Propellant and Exothermic Compositions", Interscience, NY (1959), 135–46 (19 refs)

Gas-Liquid Chromatography. See under Gas Chromatography

Gas Liquor. Same as Ammoniacal Liquor, briefly described in Vol 1 of Encycl, p A305-L

Gas, Mond. See under Gas Producer

Gas Munition. Munition such as bomb, projectile, pot, candle or spray tank contg a chemical agent (See Vol 2 of Encycl, pp C165-R to C178-R) and means of release
Ref: Glossary of Ordn (1959), 133-R

Gas, Natural. Any gas issuing from under the earth's crust thru openings or bored wells, may be called natural gas. The most important natural gas is that consisting of a mixture of hydrocarbons. Such gas is often found in and near coal or petroleum deposits in many countries, including the USA. Many gas wells do not yield oil, but an oil well always produces gas and oil. Such a gas is known as "casinghead gas" (Ref 1a, p 275)

Typical composition of an American natural gas is: methane (with some ethane, propane etc) 92, hydrogen 3, illuminants 3 & nitrogen 2. Heating value is 1000 BTU per cu ft. Volumes of air necessary to burn one vol of gas are 9.73. Riegel (Ref 1a, p 271) gives the following compn: methane 90.0, ethane 8.8 & N_2 1.2. Total combustibles 98.8 and BTU 1100 per cu ft

Some natural gas, eg, that issuing near Sicilian volcanos, contains sulfur ingredients as a chief ingredient. This gas may be used for the preparation of sulfur

Natural gas has been known for many centuries. For instance, near Baku, Caucasus, a temple of a religious sect who "worshipped the fire", existed long before the Russians arrived there. The fire was produced by the burning of natural gas. The first commercial use of the gas in the USA was in 1820 at Fredonia, NY, when a few buildings were

supplied from a nearby shallow gas well. From that time until about the last part of the 19th century, only a small quantity of gas was used. After that the consumption of natural gas began to grow, reaching about 500 billion cu ft in 1910. In 1964 about 15 trillion cu ft were produced and the industry is still growing. At present, about 30 states have known deposits of natural gas
Refs: 1) L. Stotz, "History of the Natural Gas Industry", RubbinsPubgCo, NY (1938) 1a) E.R. Riegel, "Industrial Chemistry", Reinhold, NY (1942), pp 271, 275 & 276 2) R.N. Shreve, "Chemical Process Industries", McGraw-Hill, NY (1945) 3) H.M. Smith & W.C. Holliman, "Utilization of Natural Gas for Chemical Products", USBureauMines, Inf Circular **107347** (1947) 4) J.H. Perry, Ed, "Chemical Engineers' Handbook", McGraw-Hill, NY (1950), pp 906, 1575–1596, 1710 & 1721–1722 5) R.L. Huntington, "Natural Gas and Natural Gasoline", McGraw-Hill, NY (1950) 6) Merriam-Webster, "Unabridged Dictionary", Springfield, Mass (1951), pp 1036 & 1631 7) Encyclopaedia Britannica (1952 ed), Vol **16**, p 163 8) R.E. Kirk & D.F. Othmer, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **10**(1966), pp 443–462, D.E. Holcomb 9) CondChemDict (1971), 608-L

Gas, Noxious. Any poisonous (toxic) gas or a gas with strong unpleasant odor
Ref: Hackh's Dict (1944), 368-R

Gas Oil. A liquid petroleum distillate with a viscosity and boiling range (450–800°F), betw kerosene and lubricating oil (Ref 2, p 528-L)

Under the title **Oil Gas** of the same ref, but p 825-R, is defined a gas made by the interaction of oil vapors and steam at high temps by the method used for prepn of *water gas*. A typical analysis of oil gas is CO 10.4, hydrogen 47.6, methane 27.0, CO_2 4.6, illuminants 4.2, oxygen 0.4 & nitrogen 5.8%. BTU/cu ft 554

Accdg to Riegel (Ref 1), compressed oil gas is known as **Pintsch Gas**
Refs: 1) Riegel, "Industrial Chemistry" (1942), 277 2) CondChemDict (1961), p 528-L (Gas Oil), p 825-R (Oil Gas); 8th edit (1971), 411-R (Gas Oil) & 642-L (Oil Gas)

GASOLINE (Gasolene or Petroleum Benzine) (Petrol by the British; Petroleumessenz or Petroleumbenzin in German; Benzine de petrole, Petrole or Essence in French; Bencina or Gasolina in Spanish; Benzina in Italian; Neftianoy benzin in Russian)

Gasoline is a colorless, transparent, highly volatile and combustible liquid with a density of about 0.75; used as a fuel in internal combustion engines, as a fuel in liquid rocket propellants, in Napalm Flame Throwers and for making rubber. Originally, gasoline was obtained by fractionating crude oil and comprised the distillate boiling between about 60° and 200°. This is now called "straight run gasoline". The composition of this varies with the type of petroleum used, and may be divided into paraffinic, naphthenic and aromatic

There is also the so-called "casinghead gasoline", which is "natural gasoline" obtd by the recovery of the normally liquid hydrocarbons which are contained in the natural gas present in oil wells, and from some oil wells which give natural gas not associated with petroleum. Present-day American gasoline is a blend of natural gasoline, straight run gasoline and the products of various processes, such as *cracking* and *alkylation*. The Germans obtd nearly all of their gasoline during WWII from coal. Some European countries use gasoline contg as much as 40% alcohol as motor fuel

Cracking gas was briefly described in Vol 3 of Encycl, p C552-L, while cracking processes, such as of Dubbs and of Houdry are described in Ref 4, pp 429–31. See also Ref 9, p 528-R

Alkylation process for the production of high-octane gasoline is described in Ref 4, p 442 & Ref 9, p 41-L

Other methods for prepn of high-octane number gasolines are described in Ref 4, pp 441–42

Method of prepn of gasoline by vapor-phase hydrogenation of lubricating oil is described in Ref 4, p 437

Refs: 1) V.N. Ipatieff et al, IEC **27**, 1067 & 1364(1935) 2) F.E. Frey & H.J. Hepp, IEC **28**, 1439(1936) 3) L.A. Munroe & E.R. Gilliland, IEC **30**, 58(1938) 4) E.R. Riegel, "Industrial Chemistry", Reinhold, NY (1942), 275, 424 & 429–34 5) J.B. Godwin & R.B. Kennedy, PetrEngr **16**(8), 176(1945) 6) E.E Pettibone, PetrEngr **16**(9), 149(1945) 7) E.J. Houdry, USP 2414812(1947) 7a) F.D. Rossini, AnalChem **20**, 110–21 (1948) 8) R.E. Kirk & D.F. Othmer, Eds, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **10**(1966), pp 463–498: J.C. Lane, "Gasoline and Other Motor Fuels" (31 refs) 9) CondChemDict (1961), 528-L;. (1971), 411-L

Gasoline, High Octane Number. It has been required that present *aviation gasoline* must be antiknock, which means that its octane number must be as high as 100. The octane number is the rating of gasoline as to its antiknocking properties on the basis of a standard sample consisting of iso-octane (2,2,4-trimethylpentane) and n-heptane mixed in various proportions. Heptane tends to knock, while iso-octane has marked anti-knock properties. By mixing the two in all proportions, a series of fuels is obtd which covers the whole scale of possible mixts. For example, a gasoline has an octane number 75 if, on compressing it in a standard engine, it begins to knock with the same compression ratio which causes a mixture of 75 parts iso-octane and 25 parts of n-heptane to develop a knock in the same engine under the same conditions (Refs 1 & 2)

Accdg to Riegel (Ref 1, p 441), the 100-octane fuel for aircraft can be prepd by mixing: iso-octane 40, aviation gasoline (74-octane number) 45 & iso-pentane 15% with 3cc tetra-ethyl lead. The iso-pentane supplies the volatility at low temps which the iso-octane lacks

The term "aviation gasoline" indicates the gasoline which becomes the base of the aircraft fuel. It is either "straight-run" or "cracked gasoline", prepd by the Houdry process. Its boiling range, 100°F(37.78°C) to 249°F(120.56°C), is lower than that for std gasoline

Refs: 1) E.R. Riegel, "Industrial Chemistry", Reinhold, NY (1942), 441 2) CondChem-Dict (1961), 822-R; (1971), 411-R

Gasoline, Polymer. A gasoline produced by polymerization of low molecular wt hydrocarbons, such as ethylene, propane, and butenes. It is used in small amts for blending with other gasolines to improve their octane number

Ref: CondChemDict (1961), 528-R; (1971), 411-R

Gasoline, Reformed. A high-octane gasoline obtd from low-octane gasoline by heating the vapors to a high temperature or by passing the vapors thru a suitable catalyst

Ref: CondChemDict (1961), 528-R; (1971), 411-R

Gasoline, Straight. Gasoline produced from petroleum by distillation, without using cracking or other chemical conversion processes (See also under GASOLINE)

Ref: CondChemDict (1961), 529-L; (1971), 411-R

Gas, Petroleum. See Gas, Oil

Gas, Pintsch. Compressed Oil Gas

Gas (or Vapor) Pockets (Bubbles) in Liquid and Solid Explosives.

The presence of minute gas or vapor

pockets (bubbles) in expls renders them more sensitive to impact, friction and initiation. This is due to adiabatic compression, which takes place in these bubbles on impact etc, resulting in the evolution of a large amount of heat and increasing the temp in the neighborhood of the bubbles. This heat causes the deton of the expl surrounding the bubbles, which means that these bubbles are the spots where initiation takes its origin. This theory originated by F.P. Bowden et al is described in Vol 2 of Encycl, p B127 under "Birth (Initiation) and Growth of Explosion in solid and Liquid Explosives Initiated by Impact, Friction, etc (10 refs)

As an example of the practical application of the knowledge may be cited the sensitivity of freshly prepd Gelatin-Dynamites, as compared with those lying in storage. The tests were conducted before WWII at Apache Powder Co, Benson, Arizona and communicated to us by Dr I.A. Grageroff. A brief description was given under "Ageing(Aging) of Dynamites", in Vol 1 of Encycl, pp A110-R to A112-L. In addn, Dr Grageroff stated that some Gelatin-Dynamites decreased in "sensitivity to sympathetic initiation", as detd by the "gap test", from 6 inches to 1–2 inches after storage for several months. If, however, cartridges of these "aged" Gelatins were lightly rolled on a table by means of a smooth board, in such a manner as to inclose some bubbles in the cartridge, the gap sensitivity was restored to nearly normal. In order to show more definitely that the decrease in sensitivity was due to the disappearance of air bubbles, the freshly prepd Gelatins were subjected to prolonged vacuum in order to remove air bubbles. The result was the same as for cartridges stored for a long time; the gap value dropped to 1–2 inches

The theory of Bowden et al was disputed by W.A. Hargreaves (See p A111-L of Vol 1), who proposed his own theory. This theory was favored by Dr J. Mayer, who worked at Explosives Plant, Villa Maria, Argentina. If Hargreaves theory were accepted, it would be difficult to explain the "rejuvenation" of aged Gelatin Dynamites, as was done at Apache Powder Co Plant. It seems that combination of both theories could be accepted

Gas, Poisonous. Same as Gas, Toxic described in Vol 2 of Encycl under CHEMICAL AGENTS, pp C165-R to C171

Gas Pressure Cartridges (Gasdruckpatronen in Ger; Cartouches à pression à gaz in Fr; Patrones de presión a gas in Span). Gas pressure cartridges are used to power cartridge actuated devices (CAD's). A CAD is a pressure actuated mechanism using proplnt generated gases to produce mechanical action other than expelling a projectile. Se Vol 2, p C70-R for further description

Ref 1 describes German developments up to 1940 of gas pressure cartridges for airplane engine starters or for pilot ejection seats. An airplane starter, "Druckpatronen DP 50/144", was made with the base of the case of aluminum of Kal 4 with Fuse VI and also using a special slow burning cellulose for the rest of the case. The charge was a coarse grained perforated powder of 1000 cal heat content. An ejector seat cartridge, HG 34/4, contained 34g of a special powder made of a mixture of Nitrocellulose, DEGDN & Nitroguanidine. The igniter was 4g of strongly pressed Black Powder

Ref: 1) E.R. von Herz, Explosivst, 1954, 64–8 & 92–8, CA 48, 14210(1954)

Gas Pressures Developed on Deflagration of Propellants. See Vol 1 of Encycl, p XX

Refs: 1) H. Muraour, "Poudres et Explosifs", Paris (1947), 73–4 2) A.G. Gorst, "Propellants & Explosives", Oboronghiz, Moscow (1957), 56–8 3) Ibid, 2nd Edn (1972), 59–61

Gas Pressures Developed on Explosion or Detonation of Explosives

In Vol 2 of Encycl, pp B180–B184, blast effects produced in air, earth and water were described. It was stated on p B180-R that,

when a HE bomb detonates, the solid bursting charge is rapidly converted into gaseous products. This process, occurring in approx 0.0001 sec develops very high pressures and temperatures. These values vary with the chem compn of the expl but their order of magnitude is 100000 atm (700 tons/sq inch) pressure and about 3000°(5400°F) temperature. Of the total energy available from the expl, as much as half may be used to expand and break the casing (bomb body), while the remainder to compress the surrounding environment. This latter energy is responsible for blast effects. Bombs intended to be used for blast effects, were constructed during WWII of thin casing

Prior to WWII, expls used in bombs were tested by Trauzl Lead Block Method and the assembled bombs were detonated in an enclosure to determine the number and penetrating power of the bomb fragments (See Panel Fragmentation Test in Vol 3 of Encycl, p 349 and Fragment Velocity Test on p C350). As these tests did not give any quantitative values for blast effects, special tests were developed, first in England (1938) and later in the USA. A brief description of these tests and refs is given under Blast Meters in Vol 2, pp B214-R & B215-L

In expressing the blast effect of an expl, it is necessary to describe its *peak pressure* and *impulse* (See Vol 2, p B180 and Fig giving Typical Pressure-Time Record for the Blast From a Bomb)

In Vol 4 of Encycl, p D483, it was stated that one must distinguish betw *pressure produced on detonation by gases* and *total pressure developed on detonation.* It was also said that pressure can be produced by expls evolving no gases, such as by reaction:

$2Al + KClO_3 = Al_2O_3 + KCl$

but accdg to C. Dunkle the products of reaction formed at high temp of expln are gaseous KCl, AlO, Al_2O and oxygen. The Al_2O_3 does not form until the products cool. Dunkle also stated that "total detonation pressure" is also known as *stagnation pressure*, and is equal to the sum of *static pressure* and *dynamic pressure*

Accdg to Cook (quoted in Vol 4 of Encycl, p D 484-L), the detonation pressure (p_2) cannot be measured directly (at least in condensed expls), owing to its transient nature and its exceedingly high magnitude. This pressure can, however, be accurately determined by the hydrodynamic equation listed in Vol 4 of Encycl, p D484-L

A more accurate, but rather complicated formula is given on p D484-R and a rather simple formula by Dunkle (p D484-R)

Other formulas for calcn of pressure are given in Vol 3 of Encycl, p C330, Stettbacher (Ref 2, p 71), Gorst (Ref 4, p 56), Bandurin & Rukin (Ref 6, p 70) and Gorst (Ref 7)

The following calcd values of "total detonation pressures" are given in Table G5, from Vol 4 of Encycl:

Table G5

p D487	RDX	346000 at d 1.785
"	TNT	200000 " 1.64
p D492	PETN	95300 " 1.00
"	"	140500 " 1.20
"	"	195500 " 1.40
"	"	262800 " 1.60
"	Tetryl	91800 " 1.00
"	"	160400 " 1.28
"	"	218100 " 1.45
"	"	259100 " 1.61
"	PA	83000 " 1.03
"	"	120700 " 1.23
"	"	164600 " 1.39
"	"	239400 " 1.63
"	TNT	68700 " 1.00
"	"	132800 " 1.29
"	"	178000 " 1.46
"	"	216200 " 1.59

The values for calculated total pressure, given in the book of Gorst (Ref 4, p 75), are reproduced in Table G6

Table G6

Explosive	Density	Total Pressure
TNT (Trotil, in Russ)	1.59	193000
TNT	1.45	157000
RDX (Ghekso-ghen, in Russ) phlegmatized	1.62	296000
Tetryl (Tetril, in Russ)	1.61	229000
PETN (Ten, in Russ)	1.60	255000

Davis (Ref 3, p 132) gives the values (See Table G7 for gas pressures detd by exploding the materials, at densities indicated, in a small bomb and measuring the pressure by means of a piston and obturator

Table G7

Explosives	Densities of Loading		
	0.20	0.25	0.30
	Pressures in kg/cm^2		
Trinitrobenzene(TNB)	2205	3050	4105
Trinitroaniline (TNA) or Picramide	2080	2885	3970
Trinitrophenol (TNPh) or Picric Acid (PA)	2150	3055	3865
Trinitroresorcinol (TNR) or Styphnic Acid	2080	2840	–
Trinitrotoluene (TNT)	1840	2625	3675
Trinitro-m-cresol (TNCrs)	1760	2480	3360
Trinitro-m-xylene (TNX)	1635	2340	2980
Trinitromesitylene (TNMes)	1470	2200	2780
Trinitronaphthalene (TNN)	–	2045	2670

This Table shows that TNB is the most powerful explosive among the nitrated aromatic hydrocarbons. One amino group, as in TNA, reduces its strength and so does one hydroxyl group as in TNPh (PA). Two hydroxyl groups have less effect than one methyl group as in TNT and TNR is a stronger expl than TNT. One methyl group reduces the strength of TNB less than do two methyls in TNX, and the weakest is TNMes with three methyls. TNT is a stronger expl than TNCrs, which differs from it in having an hydroxyl group. The weakest of the above expls is TNN because it contains more C's than any of them

The Table on p 157 of Davis (Ref 3) gives the following values for nitrated derivatives of naphthalene at density 0.3: MNN 1208, DNN 2355, TNN 3275 and TeNN 3745. The value given here for TNN is higher than those given on p 132 of Ref 3

The values given in Ref 3 on p 169 for TNR and TNPh, 2260 & 2350, respectively, at density 0.2 are higher than those given on p 132

The following values are given in Table of Ref 3, p 172: TNPh 2310, 2350 & 2210 at d 0.2 and 3230 at d 0.25; Trinitroanisol – 2222, 2250 & 2145 at d 0.2 and 2850 at d 0.25; Trinitrophenetol – 1774 at d 0.2, 2490 at d 0.25 and 3318 at d 0.30

In Table on p 175 (Ref 3) are given pressures for TNA 2356 at d 0.20 & 3110 at d 0.25; for Tetryl at d 0.20 – 2423 and at d 0.25 – 3243

In Table on p 182 (Ref 3) are given the following pressures at d 0.3 and temps indicated: Tetryl 4684 at 2911°C, TNPh 3638 at 2419°, TNT 3749 at 2060° and TNB 3925 at 2356°C

Devices for experimental determinations of pressure of gases developed on expln or detonation are described in Vol 3 of Encycl, pp C330-R to C345-R, under "Closed Bomb(or Vessel) and Instruments for Measuring Pressures Developed by Explosives or Propellants". Vertical bomb of Dolgov used in Russia instead of horizontal Bichel Bomb is described by Yaremenko & Svetlov (Ref 5, p 75 and Fig 34). This bomb was also used for determination of volume of gases developed on expln. Manometric bomb used in Russia for dem pressure of gases developed on deflagration of propellants is described by Gorst (Ref 4, pp 56–8 and in Figs 10, 11, 12, 13 & 14) and his new edition (Ref 7)

A detailed description of Bichel Closed Bomb employed in 1931 at USBurMines was given on pp 85–87 of Ref 1 (under the name Bichel Gage), together with description of procedure used at that time. The Fig shown on p 86 of Ref 1 is just a photo of exterior view. A clearer idea of construction can be obtd from Fig of Vol 3 of Encycl, p C331-R and from Fig on p C332-L

Procedure described in Ref 1, pp 84–95 was used to determine the *maximum pressure a mining explosive would exert if exploded in a space that it fills completely, and all of the heat set free by the chemical reactions that take place being retained by the products of explosion.* Briefly it means: "maximum pressure of the explosive in its own volume".

Equipment. At the time of publication of Ref 1 (1931), there were two Bichel Closed Bombs at BurMines, each of them made of strong cast steel. The **No 1** apparatus had an interior capacity of 15 liters, while the **No 2** was of 20 liters. Their walls were 12.5cm thick. The heads of their cylinders were provided with lead gaskets, which were secured in place by 12 heavy stud bolts and an iron yoke. For exhausting the air there was a tube inserted in a hole bored in an upper segment of each cylinder (See Fig on p C332-L of Vol 3 of Encycl), near one end. Exhaust tube of each cylinder was connected with a rotary, vacuum-air pump, driven by a 2-HP motor and provided with a valve which excluded the outside air after the desired vacuum was reached (usually 50±5mm of Hg). In the upper segment of the cylinder, on the end opposite the exhaust tube, was an opening for the insulated plug that provided a means of conducting the electric current to the electric detonator and prevented air entering the cylinder while it was being exhausted. It also prevented escaping of gases after the expl was fired and while considerable pressure existed in the cylinder. A 3rd perforation, made in the top of each cylinder, allowed a properly glanded tube to be inserted in this aperture. The tube was provided with a piston 0.3937cm diam, which could be moved up and down within the tube and was resisted by one of the four springs provided for this purpose. These springs exerted different pressures. They were marked 15mm 4kg, divide by 0.60mm = 1kg/sq cm; 10mm, 6kg divide by 0.40mm = 1kg/sq cm; 8mm, 8kg divide by 0.32mm = 1kg/sq cm and 10kg divide by 0.24mm = 1kg/sq cm. It was desirable to select a spring that gave a curve 35±10mm high on the paper or card attached to the drum described below. A stylus was mounted on one end of lever, the other end of which was fastened to the upper end of the stem of the piston in such a manner that while free to move vertically, the stylus should remain in the same vertical plane as long as the gases generated in the cylinder exerted pressure upon the piston. Attached to the upper part of each apparatus and to the rear of the piston rod bearing the stylus, was a support carrying a recording drum with indicator paper ribbon attached. The drum was mounted to rotate horizontally about a vertical axis. The mechanism by which the drum was rotated was geared to an electric motor that ran at a constant speed, that could be accurately measured by means of a speed counter. Hence, the curve drawn on paper ribbon could be resolved with accuracy into its components of horizontal translation of the paper and vertical elevation of the stylus at the different periods thruout the expln. Usually the drum rotated at a speed of 412rpm. In the center of each apparatus was a small wire support upon which the cartridge of expl was laid, thus preventing the destructive effects on the walls of the cylinder that an expl exerts when it is detonated in contact with a surface

Apparatus No 2, being constructed in the same manner and fitted with similar appliances as No 1, was further provided with four solid steel cylinders which served to reduce the volume of the chamber while varying its cooling surface. Diameters of cylinders were 17.78cm, while heights were 20cm for one and 6.67cm for each of the remaining three. While Bichel apparatus No 1 had a cooling surface

A=3914sq cm, apparatus No 2, with large solid cylinder inside had a surface B= 6555sq cm and a surface C=7624sq cm when the large solid cylinder was replaced by three small ones

Procedure for Determining Pressure of Explosive in Its Own Volume. The chge required to test a permissible expl at BurMines was 200±5g (of expl itself), to which was added the proportional amt of the wrapper. After removing the head of the Bichel cylinder and wiping it dry inside, the chge of expl, provided with a No 6 electric detonator was placed on the wire support. One leg of detonator wire was fastened to the wire that passed thru the insulated plug on the upper segment of the cylinder, while the other leg was grounded to the gage thru the iron support with which it was in contact. The head was then placed on the open end of the cylinder and secured to it by nuts screwed on to stud bolts, which were fastened to that end of the cylinder. The cylinder was then exhausted until the vacuum reached 50±5mm, the difference betw the barometer and manometer readings. After bringing the temp of the gage to 22±5°, the charge was fired by electric current and then the cylinder allowed to cool while the indicator continued to function

As was mentioned above, the gage spring was selected to produce a curve 35±10mm at maximum pressure, but if the 1st trial failed to produce such a curve, another spring was tried, etc until a satisfactory one was found. The spring chosen as satisfactory was then used in all of the trials of the same expl under test. A careful record was made of the spring used, and its effect was noted in each experiment made

After completion of each test, using cooling systems A, B and C, the heights of curves on recording paper were measured and the pressure, P, developed in the gage was computed to the nearest 0.01kg/sq cm by dividing the height of the curve in mm by the value for the particular spring used (one of the four listed above). Each value was expressed to the nearest 0.01mm as being equivalent to 1kg/sq cm

If value A is the pressure developed with cooling system A, value B with the cooling system B and value C with the cooling system C, then pressure shown on the curve is:

$$P = 1911A + 0.5B - 1.411C$$

This pressure P, divided by the charge density (defined as the volume of the chge divided by the volume of the bomb), gives the maximum pressure of the expl in its own volume after elimination of cooling-surface influence

To simplify the computing of the pressure (M) in its own volume, the formula:

$$M = \frac{VPS}{W}$$

was adopted. Here V=volume of the Bichel cylinder (15000cc); P=pressure obtd from the curve; S=apparent specific gravity of the cartridge as detd in the physical examination; and W=weight of the chge in grams. M was expressed to the nearest kg/cm. For expressing in psi, it is necessary to multiply kg/sq cm by 14.2232

Determination of Pressure of Low Explosives. In testing low expls, such as Black Blasting Powder, the charge was usually 300g, plus 2.5g of newspaper wrapping per 100g of BkPdr. The chge was fired by means of an electric igniter. Firing could be done either in vacuum or with air under atmospheric pressure. The measurement of pressure from the indicator cards was as follows: The maximum pressure was usually reached in about one revolution of the drum and continued for approx another revoln. The number of revolns and fractions thereof was measured from the firing to the point where the pressure began to fall owing to the cooling of the gases. At this point the maximum pressure was scaled off, and the average fall in pressure thru three succeeding revolns was added. This sum gave the corrected normal pressure

See also "Gases Produced on Explosion or Detonation of Explosives" and also "Gas Volumes Developed on Explosion or Detonation of Explosives"

Determination of pressure using Crawshaw-

Jones Apparatus was described in Ref 1, pp95–99
Refs: 1) Ch.E. Munroe & J.E. Tiffany, "Physical Testing of Explosives", USBurMines Bull **346**(1931), pp 84–90 2) A. Stettbacher, "Schiess- und Sprengstoffe", Barth, Leipzig (1933), p 71 3) T.L. Davis, "The Chemistry of Powder and Explosives", Wiley, NY, Vol 1 (1941), pp 132, 157, 169, 172, 175 & 182 4) A.G. Gorst, "Powders and Explosives", Oboronghiz, Moscow (1957), pp 56 & 75 5) N.E. Yaremenko & B.Ya. Svetlov, "Theory and Technology of Industrial Explosives", PromstroyIzdat, Moscow (1957), pp 75–6 6) M.K. Bandurin & A.G. Rukin, "Collection of Problems on Theory of Explosives", GosIzdatOboronProm, Moscow (1959), p 70 7) A.G. Gorst, "Powders and Explosives," Mashinostroyeniye, Moscow (1972), pp 59–61 & 79

Gas, Producer and Mond Gas. A gas obtd by burning coal or coke with a restricted supply of air. It is made in steel, brick-lined vessels such as 10ft in diam and 12ft in height (See p 274 of Ref with Fig 100). Any low-grade fuel may be partly burned, while steam in addn to air is sent into fire continuously, while fuel is dropped into apparatus at constant rate. The resulting producer gas passes out continuously, while the ashes are removed continuously by a sweeper in the water seal at the base. Numerous types of producers are on the market

The reactions are the same as those which take place in a water-gas plant (qv); but in producer gas there is contained all the nitrogen entering with air for combination; hence, total combustible constituents of producer gas and the heat value per volume are lower than those of water gas

The air-steam mixture (approx 7 vols of air for 1 vol of steam) is forced into app by means of an injector. The amt of steam used for 1 lb of coal gasified lies betw 0.3 and 0.5 lb. The vol of gas produced from bituminous coal is about 60 cu ft; semibitumiinious yields 30 and lignite 28 cu ft

The so-called **Mond Gas** is producer gas made at low temp so that the amount of ammonia in the coal may be conserved and later recovered

A typical compn of producer gas is given on p 271 of Ref: CO_2 5.2, ethylene 1.0, oxygen 0.2, CO 26.3, hydrogen 14.4, methane 1.8 & nitrogen 51.1. Total combustibles 43.5 and BTU per cu ft 155.6
Refs: 1) E.R. Riegel, "Industrial Chemistry", Reinhold, NY (1942), pp 170–71 & 174–75 2) CondChemDict (1971), 731-L

Gas Pyrofax. It is propane, C_3H_8 (bp –42.5°C), compressed in steel cylinders which are transportable
Ref: Riegel, "Industrial Chemistry", (1942), 277

Gas Shells. No casualty-producing gas shells were used until near the end of WWI (June 1915) when the Germans brought out their K shell. The allies began firing such shells in Jan 1916. From then on the percentage of gas shells used on both sides steadily increased. By far the greatest number of all gas shells used in WWI was fired on the Western Front, next came the Eastern Front, and the Austro-Italian Fronts followed

Unlike HE shell which had been fully developed and were std munitions 30 yrs before WWI, gas shells had to be hastily improvised & developed under stress of war conditions. Many of the early gases used in artillery shell proved unsuitable or were not adapted to field conditions. Thus, out of more than 50 chem substances loaded into artillery shell, only 4 or 5 proved effective in battle. Often the efficiency of a shell could be definitely ascertained only after a large number of rounds had been fired. For example, the French Vincennite shell was filled with a mixt of hydrocyanic acid & arsenic trichloride, which had a marked toxicity in lab tests. Yet, after 4 million shells were filled with this mixt it was not an effective gas under battle conditions

In addn to inefficient toxic gases, there were a large number of shells of the lacrimatory & irritant types not intended to produce casualties, but to harass the enemy. When the various noncasualty-producing shells are subtracted from the total, a very high casualty power for successful types, such as phosgene & mustard shells, is disclosed. Considering the seven countries engaged in chem warfare during WWI (Austria, England, France, Germany, Italy, Russia & USA) one casualty was produced for each 100 rounds of nongas artillery ammo, but one casualty for each 45 rounds of toxic-gas shells resulted

Much has been written concerning the horrors of gas warfare and the cruel & inhuman consequences resulting. Some of these alleged horrors were pure propaganda deliberately disseminated to influence world opinion. In general, gas caused less suffering than wounds from other weapons. Chlorine, the first gas used, did cause strangulation & high mortality, but when troops were supplied with gas masks, chlorine became the most innocuous of toxic gases and was the least feared by both sides

Two other principal lethal gases, phosgene & chloropicrin, in high concentrations caused instant collapse, but no suffering. One of the most striking evidences of the effectiveness of gas shells & chemical warfare agents was their rapidly increasing use as WWI progressed. There is no doubt, had the war continued, the campaign of 1919 would have been largely a chemical war. This phenomenal rise in the use of chemicals from unknown obscurity in 1915 to the position of a military agent of the first magnitude in 1918 was without parallel in the history of warfare up to that time
Ref: A.M. Prentiss, "Chemicals in War", McGraw-Hill, NY (1937), 647–84

Gas, Tar. See Coal Tar and Coal Tar Pitch in Vol 3 of Encycl, pp C379-R to C380-R

Davis (1941), pp 129–32 gives a detailed description of "Utilization of Coal Tar"

Gas Testing Apparatus Suitable for Detecting the Presence of Explosive or Combustible Gas in Air is described in
Refs: 1) Mine Safety Appliances Co, BritP 520993 & CA **36**, 1168 (1942) and
2) R.E. Hartline, USP 2279397 & CA **36**, 5352 (1942)

Gas, Toxic. Same as Gas, Poisonous described in Vol **2** of Encycl, under CHEMICAL AGENTS, pp C165-R to C171

Gas Turbines. A heat engine that converts some of the energy of fuel into work by using gas as the working medium and that commonly delivers its mechanical output thru a rotating shaft. The sequence of thermodynamic processes consists basically of compression, addition of heat in a combustor, and expansion thru a nozzle. The flow of gas is continuous during these thermodynamic changes
Refs: 1) M.J. Zucrow, "Principles of Jet Propulsion and Gas Turbines", Wiley, NY (1948) 2) E.F. Flock & C. Halpern, "Bibliography of Books and Published Reports on Gas Turbines, Jet Propulsion and Rocket Power Plants", USNatlBurStandardsCircular **509** (1953) (64pp) and a Supplement (1954) (110pp). It covers period up to Jan 1954
3) D.N. Lapedes, "McGraw-Hill Encyclopedia of Science & Technology", Vol **6**, McGraw-Hill, Inc, NY (1971), pp 65–71

Gas Volumes Produced on Explosion or Detonation of Explosives. The volumes can be determined either theoretically by calculation or experimentally

Determination of vol by calcn is described in Stettbacher (Ref 2, pp 68–9), Gorst (Ref 3, p 54), Yaremenko & Svetlov (Ref 4, p 74) and Bandurin & Rukin (Ref 5, p 69)

If formula of deton (or expln) of an individual expl like NG or TNT is known, it is easy to calculate V_o, which is **specific volume** of gases in liters, produced on expln of 1kg of expl, calcd to 0°C & 760mm with water vapor

For example, for NG Stettbacher (Ref 1) gives:

$4C_3H_5(ONO_2)_3 = 12CO_2+10H_2O(vapor)+6N_2+O_2$

4 x 227.06 (12 + 10 + 6 + 1) x 22)412

908.24 = 649.95 liters or 715.6 liters for 1kg

Value, V_o, for NG given in Ref 4, p 75 is 716 liters/kg

For calcg of volume for water liquid if vol of water gas is known, first calculate the volume at 100°C according to $V_{100} = V_T(373°K/T)$ where V_T is the known volume at temp T(°K). Then divide V_{100} by 1672 (which is a combination of the shrinkage factor of 1603 for steam at 100°C going to water at 100°C, and of the shrinkage factor of 1.043 for water at 100°C going to water at 0°C). The correction for steam being non-ideal is minor

Volume, V_t, at temperature , t, produced on expln can be calcd by Gay-Lussac's Law, known in the US as Charle's Law, also called Dalton's Law:

$$V_t = \frac{V_o(273+t)}{273} = \frac{V_o T}{273} = \frac{715.6x(273+4215)}{273} = 11751 \text{ liters/kg}$$

If V_o for TNT at 0° & 760mm Hg is 740.6 liters/kg, it equals 9015 liters at its temp of deton 3050°C (Ref 2, p 69)

If compn of the expl mixture is known and its equation of expln can be derived, calcn of specific volume V_o can be done in the same manner as described above

For example, Russian Ammonit No 6 cont AN 79 & TNT 21% (Ref 4, p 68) can be written as $C_{6.5}H_{44.1}O_{35.1}N_{22.6}$, and its gases of detonation: $6.5CO_2+22.H_2O+11.3N_2$

This gives: $V_o = 22.4(6.5+22.1+11.3) = 893.6$ liters/kg (Ref 4, p 74)

If compn of gases produced on expln of an expl mixt is unknown, the gases must be removed from the apparatus like Bichel Bomb or Crawshaw-Jones Apparatus (both described in Ref 1, pp 84–95), collected in a gasometer and then analyzed by one of the methods listed here under "Gas Analysis", like that of Orsat. The device used in Russia for detn of volume of gases is the vertical bomb of Dolgov. Its description and procedure are described in Ref 4, p 75. Interior capacity of these bombs is between 15.5 and 50 liters

The values of V_o experimentally detd in Dolgov's Bomb and listed in Ref 4, p 76 are reproduced here in Table G8

Table G8

Explosive	Density g/cc	Specific Volume V_o	
		H_2O liq	H_2O gas
Trotil (TNT)	1.5	610	750
Tetril (Tetryl)	1.55	620	740
Gheksoghen (RDX)	1.5	700	890
TEN (PETN)	1.65	550	790
Ammonit No 6 (AN 79 & TNT 21%)	1.3	430	890

Note that volume of gases detd by calcn for Ammonit No 6 was 893.6 liters/kg with H_2O gas (Ref 4, p 74) and for TNT as reported by Stettbacher (Ref 2, p 69) was 740.6 liters/kg

Following values for V_o are given in Ref 5: p 71 – NG 716.8; p 72 RDX 922.6; p 72 NC $[C_{24}H_{29}(ONO_2)_{11}]$ 862.3; p 73 MF 236.12; p 74 Blasting Gelatin 716.8; and p 74 PA 784 liters/kg

Experimental Procedure for Determination of Volume of Gases as Practiced at Bureau of Mines. The method described below is used as a permissibility and suitability test of mining expls. For this purpose a 200g chge of the expl and its proportional amt of the original wrapper, as found for a 1¼-inch diam cartridge, is fired in Bichel App No 1. The method followed in prepg and firing the chge is the same as that employed in making the detn of the pressure described here under "Gas Pressures Developed on Explosion and Detonation of Explosives". It was required, however, that the Bourdon type gage be substituted for the regular piston indicator mechanism. The Bourdon gage was connected to the chamber of the bomb after the shot was fired. Before firing the chge, the bomb was evacuated until a vacuum of 50±5mm was obtd. The vacuum data used were the differences betw the barometer reading for the atm and the manometer reading for the gage. In order that the operator could be assured of obtg vacuum and that there were no clogged lines, the valve connection for the pressure gage on top of the bomb remained open until

after the vacuum pump had been started and air was sucking thru the valve. The chge was fired by means of a No 6 electric detonator imbedded in the chge within the bomb. Five minutes after the shot was fired the stopcock which intervenes betw the bomb and the Bourdon gage was opened and the pressure noted. When the gage reading was obtd the gaseous products within the bomb had cooled down to approx room temp. Then the volume of gases and vapors at 0°C and at pressure of 760mm Hg was calcd from the data obtd, which comprise bomb temp, bomb pressure, barometric pressure and volume of the bomb (approx 15 liters)

Taking Samples of Gases and Vapors for Chemical Analysis. One half-hour after the shot was fired a sample of the gases and vapors was taken thru the valve to which the air pump was attached. The gases were allowed to escape from the bomb slowly by cracking the valve, and a 200cc sample was thereby collected over mercury of the aspirator during the entire time of the escape of the gases. In collecting a sample the mercury of the aspirator was allowed to run out, and the gases and vapors that followed were drawn off until the pressure within the bomb was reduced to that of the atmosphere; therefore, the sample of gases and vapors taken was a differential one

The liquid and solid products of the reaction were collected and measured after the head of the cylinder had been removed, the liquid products being drawn off into a measuring vessel and the solid products scraped out and weighed. Since secondary reactions might take place as soon as the air struck the products, the material collected might not be identical with that immediately resulting from the reaction; however, no better method appears to have been devised

The gaseous products were analyzed by means of an Orsat apparatus and then entered upon special form, under "Analysis of Gases" (Volume per cent). An example of calcn is given in Ref 1, p 94
See also "Gases Produced on Explosion or Detonation"

Crawshaw-Jones Apparatus. It was recognized that the Bichel apparatus possesses several defects, the chief one being that the conditions under which the explns take place in a Bichel bomb differ from the conditions under which the explns are fired in actual mining operations. This difference lies chiefly in the following facts: In mining, the chge of expl is confined closely within the borehole by stemming, and, when the chge is fired, the products of expln are liberated, thru broken down coal or rock, promptly into atmosphere, where they are free to expand rapidly to an unlimited extent and, therefore, to cool quickly. In the Bichel bomb, however, the chge of expl is practically unconfined when fired, but its expln products are confined within the walls of the bomb where they cool down with relative slowness. Secondary reactions take place, meanwhile, betw the components of the primary expln products, with the result that the cooled products recovered may differ from those produced by the reactions which took place in the expl during the expln and those liberated by the expln in actual mining practice

With a view toward correcting these errors and making laboratory practice conform to field practice, so far as possible, J.E. Crawshaw and J.W. Jones devised and developed the Crawshaw-Jones Apparatus at the Explosive Experiment Station, Bruceton, Pennsylvania

Since the C-J apparatus is not described in the literature and since BurMinesBull (Ref 1) is out of print we are including here the description given on pp 97–99 of Ref 1

This apparatus is illustrated in Fig G13. For convenience in description, the sketch may be regarded as divided into two principal parts

Part 1 consists of:

1) Firing cannon, *a*, a forged-steel cylinder 24 inches in diam and 36 inches long with a borehole 2¼ inches in diam and 21½ inches long. It is mounted on a movable truck so that it can be moved to and from pipe *b* for loading the cannon and cleaning chamber *b*

2) An expansion chamber, *b*, which is a piece of 8-inch extra-strong lapwelded wrought-iron pipe 10 feet long, flanged at each end and

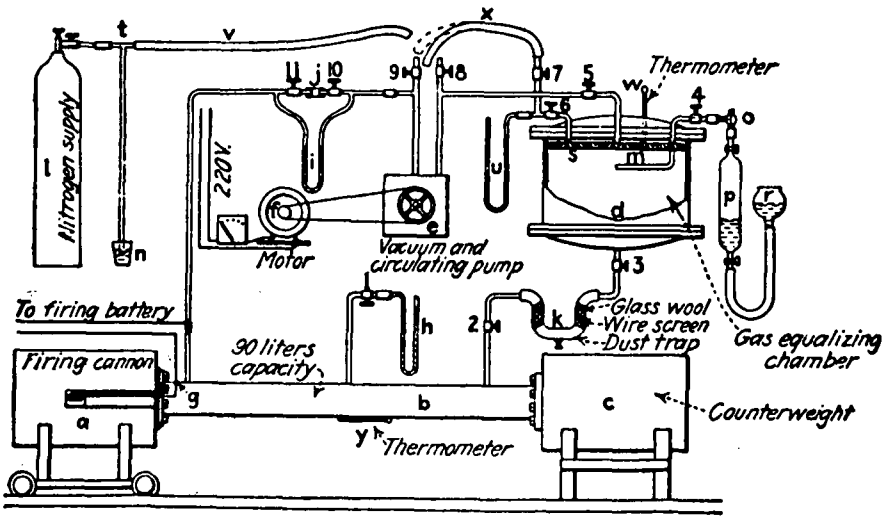


FIG G13 SKETCH SHOWING DETAILS OF CONSTRUCTION OF CRAWSHAW-JONES APPARATUS AND ACCESSORIES

bolted to the cannon and counterweight *c* by twelve 7/8-inch steel bolts

3) Counterweight *c* of the same dimensions as the cannon *a*, to stabilize the apparatus when the explosive is fired from *a*

Part 2 consists of the pressure-equalizing, mixing, and sampling devices. By partly evacuating equalizing chamber *d* before firing at *a* the pressure in expansion chamber *b* can be released into it immediately after a shot with a resultant pressure in the whole apparatus of slightly more or slightly less than atmospheric, which lessens any tendency to leak during the cooling and mixing of the gases. Pump *e* is used to evacuate equalizing chamber *d*, which is used for mixing the gases and sweeping out the entire apparatus after each test

Charges of 150 to 200 grams are used in each test. This charge of explosive gives detonation products of a good degree of concentration and does not produce excessive pressures within the apparatus at the instant of detonation, and the effects of the products from the detonator can be disregarded. Moreover, charges of this size can be packed in the borehole in the same manner for each shot.

Test Procedure. The procedure of testing is as follows:

The stud bolts which hold the firing cannon *a* to pipe *b* are unscrewed, and the cannon is rolled back out of the way. A scraper is used to clean the pipe of stemming material and foreign material from the previous trial, and the first 3 feet of the pipe next to the firing cannon is wiped out thoroughly. The borehole of the firing cannon is then cleaned of residue from the previous trial, and the explosive and electric detonator are charged in the cannon according to the desired method

Stemming is then inserted in the borehole and tamped firmly against the expl. The ends of the legs of the electric detonator are then cut off at the proper length, and one leg is fastened to a screw in the face of the cannon

When the apparatus has been cleared of gases from the previous trial the cannon is rolled close to the pipe and the other leg from the electric detonator is joined to firing plug *g*. The cannon is then rolled against pipe *b* and made gastight by screwing down the stud bolts. Valves 2, 3, 4, 7, 8 and 10 are closed and valves 5, 6 and 9 (tube *v* being disconnected from 9) opened; the pump is then started, thus evacuating the air from chamber *d*. When a sufficient vacuum is produced in chamber *d* to take care of the

volume of gas it is estimated will be liberated by the expl, valves 5 & 9 are closed, the pump is stopped, and valve 11 is closed. The charge of expl is detonated by means of the electric detonator fired by the aid of a push-down battery, and the gases liberated into pipe *b* by the expl are quickly released into the chamber *d* by opening valves 2 & 3. Valves 5, 10 & 11 are then opened and pump *e*, which circulates the gases thru the pipe, equalizing chamber, and pipe connections, is operated. Tests have shown that five mins of circulation is sufficient to mix the gases thoroughly. At the end of five mins the pump is stopped, and the pressures on manometer *b* and *u* and the temp on thermometers *y* and *w* are read. The pump is then operated five mins more and the temp and pressure readings again noted. These last values are used in the calculations of the results

Sampling gas. – A sample of the gas is then withdrawn for analysis by means of sampling tube *p* and mercury leveling bottle *r*. Cock *o* is opened to connect the sampling tube with the outside air. The cocks on the sampling tubes are opened, and mercury-leveling bulb *r* is raised, thus causing the tube to fill with mercury. When the mercury reaches cock o the cock is turned to connect with the pipe leading to the interior of the apparatus. The mercury-leveling bulb is then lowered, and thus a sample of gas is drawn out from the drum. This sample is discarded because it contains air from the connection, and a second air-free sample is withdrawn in the same manner

Analysis of gas. – The sample of gas thus obtd is then analyzed for carbon dioxide, oxygen, carbon monoxide, hydrogen, and methane on a BurMines Orsat apparatus. Oxides of nitrogen are tested for in a seoarate sample. The nitrogen is detd by difference. If the volume of the entire apparatus, the temp and pressure of the gas, and its compn as given by analysis are known, the amounts of the different constituents produced by the explosive can be computed

Accessories to apparatus. – Orifice *j* was placed in the circuit to indicate on flow meter *i* how the pump is operating in circulating the gases thru the apparatus. High rates of flow of gas are shown by large differences of level in the two columns of liquids in the flow meter

Pump *e* is of the friction-plate type and can be used for either vacuum or pressure. It is best to operate the pump with the minimum amount of lubricant that gives the vacuum desired, so that equilibrium is quickly obtd betw the gases and oil with very little effect on the gases. "Nujol", a medicinal lubricant, is used for the pump. This oil has a high fl p and has proved satisfactory for this work. A low fl p oil is undesirable because it gives off volatile constituents that will contaminate the gases from the expls. A dust trap was found necessary to prevent stemming and coal dust from getting into the drum and pump of the apparatus. It should be cleaned out after every 15 to 20 trials and the packing replaced with fresh glass wool

An attempt was made to put a thermometer well into pipe *b* for recording the temp of the gases, but in every case the wells were broken off after one or two trials by the force of the explosions. Temp readings of the gas in the pipe were obtd by placing a thermometer along the bottom of the pipe, then placing a copper shield over the Hg bulb in contact with the pipe, and covering it will insulating material. The thermometer responded very quickly to a change of temp of the pipe. Temps were recorded in two places because, 10 mins after shooting, the pipe was 1.5° to 2.0° higher in temp than other parts of the apparatus. The two values were averaged

The following typical example will illustrate the procedure for the calculation of test data:

Table G9
Sample Calculation

Volume of pressure apparatus including all parts	179.3 liters
Weight of explosives used	162.1 grams
Average temperature of cooled gases	+5.0 °C
Average manometer readings	–19 mm
Volume of space occupied by tamping in borehole	0.5 liter
Corrected volume	178.8 liters
Barometric pressure	734 mm of Hg
Corrected pressure=734–19	715 mm of Hg
Corrected temp=273+5	278 °C
Volume corrected to 0°C & 760mm pressure	165.2 liters

Table G10
Composition of gases in apparatus after test

Constituent	Per cent by volume	Gas per 100 grams of explosive, liters
Carbon dioxide	11.6	11.85
Oxygen	12.5	12.75
Carbon monoxide	3.2	3.25
Hydrogen	2.0	2.05
Methane	1.0	1.00
Nitrogen	69.7	71.00

The number of liters of gas is obtd by multiplying the percentages of gases given by analysis by the liters of gas in the apparatus and dividing by the weight of expl used

Refs: 1) USBurMinesBull **346**(1931), 91–5 2) Stettbacher (1933), 68–9 3) Gorst (1957), 54 4) Yaremenko & Svetlov (1957), 74 5) Bandurin & Rukin (1959), 69 6) Gorst (1972) (New edn of Ref 3), pp 58–9 (Full titles of these Refs are given under "Gas Pressures Developed on Explosion or Detonation of Explosives", Refs 1, 2, 4, 5, 6 & 7)

Gas Warfare Agents. See under CHEMICAL AGENTS OR CHEMICAL WARFARE AGENTS in Vol 2 of Encycl, pp C165-R to C171-L

Gas, Water and Carburetted Water Gas. Water-gas is made by passing steam thru a bed of incandescent coke or other fuel. The temp of such a fire is 1400°C(2552°F). Betw the temps of 1400° and 1000°(1832°F) the reaction $H_2O+C=CO+H_2$, which is endothermic, takes place, but if the temp drops below 1000°, the steam reacts to form CO_2: $2H_2O+C=CO_2+2H_2$ which is not desired because CO_2 has no calorific value. In order not to allow the temp to drop below 1000°, the steam is periodically stopped, and the fire is brought back to normal high of 1400° by an air blast. Coke is consumed during air-blasting, evolving not only CO but also CO_2

A detailed description of manufg water gas is given in Riegel (Ref 1, pp 271–72) together with Fig 99 representing a cross-section thru a water-gas plant

For many purposes, water gas is used as such, but for sale to municipalities, it is usually enriched with oil gas produced in a separate vessel forming part of the system. Such enriched gas is known as **carburetted water gas.** Its manuf is described on p 273 and purification on p 274 of Ref 1

Riegel gives (Ref 1, p 271) the compns of straight enriched water gases shown here in Table G11

Table G11

Components	Water Gas Straight	Water Gas Enriched by Oil
Carbon dioxide	3.7	4.25
Ethylene or other illuminants	–	10.33
Oxygen	0.5	0.71
Carbon monoxide	42.3	31.60
Hydrogen	47.9	35.38
Methane	0.6	12.40
Nitrogen	3.4	5.33
Total Combustibles	90.8	89.71
BTU per cu ft	299	574

Refs: 1) Riegel, "Industrial Chemistry" (1942), 270–74 2) Faith, Keyes & Clark (1957), 440ff 3) CondChemDict (1971), 935-L

Gathurst (Poudre de). Accdg to Daniel (Ref), one of the British powders manufd since 1895 by the Roburite Explosives Co, Ltd at Gathurst consisted of AN 84.40, DNB (Dinitrobenzene) 15.52 & moisture 0.08%
Ref: Daniel (1902), 323

Gathurst Powder. Accdg to Marshall (Ref 1), it is a British permitted expl contg AN 51, K nitrate 6, TNT 18 & NaCl 25%. Its strength by swing of Ballistic Pendulum is 2.41 inches, compared with 3.27 inches for British Standard Gelignite (60% NG)

A slightly different compn is given by Taylor (Ref 2): AN 49.0–52.0, K nitrate 5.0–7.0, TNT 16.5–18.5 & NaCl 24.0–26.0%; density 1.10, strength (% of Blasting Gelatin) 53. It was a powdery material used in moderately hard ripping and blasting in hard coal
Note: When strength (power) of Amer AN Dynamites is measured, it is compared to American Straight Dynamite contg 40% NG. Its Ballistic Pendulum Swing is 2.7 to 3.1 inches
Refs: 1) Marshall **3** (1932), 119 2) J. Taylor, "Detonation in Condensed Explosives", Charendon Press, Oxford, England (1952), 21

Gathurst Powder (Sheathed). See Vol **3** of Encycl, Table, p C452, bottom

Gattermann, Ludwig (1860–1920). A German organic chemist, noted for laboratory methods
Ref: Hackh's Dict (1944), 571-L

Gattling Machine Gun or *Organ-Grinding Machine Gun.* One of the first successful machine guns. It was invented in 1862 by an American R.J. Gattling, and used to some extent in the American Civil War (1861–1865), Franco-Prussian War (1870–1871) and Spanish-American War (1898)

The weapon consisted of ten barrels; it weighed 90 pounds and could fire up to 1 000 shots per minute
Refs: 1) J.R. Newman, "The Tools of War", Doubleday & Doran, NY (1943), p 54
2) G.M. Chin, "The Machine Gun", Vol **1** (1951), 48–63 (Gatlin Machine Guns)

Gaubitza, pronounced Gowbitza. Russian for Howitzer

Gauss, Karl Friedrich (1777–1855). A German mathematician who developed the conception of the three fundamental units: length, mass and time

In his honor was named **gauss,** the unit of intensity of a magnetic field (field strength): A magnetic field which exerts a force of one dyne on a unit magnetic pole: 1 gauss (EMU)= 0.3×10^{-10} esu. For small measurements: 1γ= 0.00001 gauss
Ref: Hackh's Dict (1944), 371-L

Gautier Receiver. A glass apparatus for collecting different samples from the same condenser during distillation in vacuum
Ref: Hackh's Dict (1944), 371-L

Gay-Lussac, Josef-Louis (1778–1850). A French chemist and physicist noted for the formulation of the gas laws. He also isolated boron and invented volumetric methods, many scientific and industrial devices (like hydrometer used for alcoholic liquid and tower used in the chamber process for the manuf of sulfuric acid to absorb the oxides of nitrogen in the crude acid produced)
Refs: 1) Hackh's Dict (1944), 371-R
2) E.R. Riegel, "Industrial Chemistry", Reinhold, NY (1942), 19 (Gay-Lussac Tower)
3) H.A.M. Snelders, ChemWeekblad **59**(43), 577–81 (1963) (Contributions to volumetric analysis)

Gay-Lussac Acid. The sulfuric acid–nitrogen oxides mixture which is the product of the Gay-Lussac tower in the chamber process for manuf of sulfuric acid. The sulfuric acid strength of Gay-Lussac acid is 60°Bé and nitrogen oxides content (calcd as N_2O_3) 1–2%
Ref: CondChemDict (1961), 529-L; (1971), not found

GDI and GDIM. Ital permissible expls listed in Belgrano (1952), p 167 without giving their compns

GDI°MT. Ital permissible expl listed in Vol 3, p C439-L

Géante (Poudre). See Giant Powders in this Vol

Geiger-Müller Counter or Tube. A common form of nuclear radiation detector, also serving for measuring the intensity of radiation such as of X-rays, γ-rays, etc. It is particularly suitable for monochromatic beams. The device usually consists of a tubular cathode with a coaxial center wire anode, filled with a gas (such as argon plus oxygen or argon plus hydrogen) at less than atm pressure. When a high voltage is impressed across the electrodes, ionizing radiation traversing the tube gives rise to conductivity pulses which may be electrically amplified and registered. Each ionizing event gives rise to one pulse, and the counter tube with its associated electrical circuitry "counts" the number of individual ionizing radiations
Refs: 1) Hackh's Dict (1944), pp 371-R & 371-L (Fig on p 372 2) H.R. Clauser, "Practical Radiography for Industry", Reinhold, NY (1952), p 13 3) CondChemDict (1961), p 529-R 4) Kirk & Othmer, Vol 17 (1968), p 72

Geissler, Heinrich (1814–1879). A Ger physicist who determined the coefficient of expansion of water and invented the so-called *Geissler Tube.* It is a partly evacuated glass tube used in the study of electric discharges thru gases and for spectroscopic examinations
Ref: Hackh's Dict (1944), p 372-L

Gel or Jel. A jelly or a solid phase of a colloidal soln, as opposed to *sol,* the liquid phase. Gel is actually a colloidal soln of a liquid (such as acetone, NG, NGc) in a solid (such as NC)
Ref: Hackh's Dict (1944), 372-L

Gels for Flame Throwers, such as "Gelgas" or "Jellied Gasoline" and "Napalm" are described in this Vol, under "Flame Throwers-Liquids and Gels" p F56

Gelamites. Trademark of Hercules Powder Co, Inc for high AN expls, introduced in 1927, which are cohesive, but only slightly plastic (semigelatinous). They are of relatively high weight strength of 65% and very good water-resistance. They can replace the gelatins, except in the most severe conditions. Used in underground mining, quarrying, construction, and general blasting. Compositions are not given
Refs: 1) J.J. Berliner & Staff, "Explosives", Pamphlet, NY (1953), pp 6 & 10 2) CondChemDict (1961), p 529-R; (1971), not found

Gelammonite No 1, Antifrost. Brit permitted expl described as Antifrost Gelammonite No 1 in Vol 1, p A468-L

Gelatin and Glue.
Gelatin, Animal is a nearly colorless, tasteless, odorless, transparent, brittle, vitroform solid, consisting of a mixture of soluble proteins of high average molecular weight. The commercial product contains 9–12%

moisture and its density is 1.3 to 1.4. Gelatin possesses the property to form a gel (jelly) in aqueous media below 35–40°. On digestion, it yields various amino-acids

Gelatin is obtained by heating collagen (the major intercellular protein constituent of the entire connective tissue of animal skins and bones) with water or dilute hydrochloric acid. Its production differs from that of glue in that the raw materials are selected, cleaned and treated with especial care, so that the resulting product is practically colorless

It is insol in cold w; swells in warm w (35–40°); sol in hot w, AcOH and a hot mixture of glycerin and water

Besides its use in foods, candies, photographic films, adhesives, gelatin capsules etc, it has been used as a binder in some explosive and pyrotechnic compositions

Glue, Animal (or Common Glue). An impure or degraded form of gelatin is obtd by action of heat and water on protein animal tissues of bones, hides or horns. It absorbs cold water with much swelling and dissolves in hot w, the soln solidifying to a jelly on cooling. Used in adhesives, gummed tapes and can be used as a binder for some expl & pyrotechnic compns

Fish Glue is obtd by heating with w the heads, fins and tails. It has weak jellying props and is generally made into *liquid glue*. For this a fish or common glue is treated with acetic, nitric, or hydrochloric acid. It has no gelatinizing props but retains the adhesiveness

Casein Glue is an adhesive prepd by dissolving casein in alkaline solns. It can be used cold

Chrome Glue is an insol product prepd by mixing common glue with Amm or K dichromate or with chrome alum. It is used as a glass cement and waterproofing material

Glycerin Glue is a flexible elastic which dissolves in glycerin

Japanese Gelatin. See Agar-Agar in Vol 1 of Encycl, p A110-L

Refs: 1) G. Stainsby, Edit, "Recent Advances in Gelatin and Glue Research", Pergamon Press, NY (1957) 2) CondChemDict (1961), p 529-R (Gelatin); p 538-R & 539-L (Glue) 3) Kirk & Othmer, Vol 10 (1966), p 499 (Gelatin); p 604 (Glue) 4) CondChemDict (1971), p 412-L (Gelatin); p 419-R (Glue)

GELATIN (Explosive). Amer name for **Gelatin Dynamite;** Brit **Gelatine Dynamite;** Ger **Gelatine Dynamit;** Fr **Gélatine dynamite;** Ital **Gelatina dynamite;** Span **Gelatina dinamita** or **Gelatina explosiva;** and Russ **Zhelatin dinamit.** *Gelatin* was invented by Swedish scientist Nobel in 1875 (BritP 4179) (Ref 3). It was a blend of NG, CC (Collodion Cotton) & 1% chalk. Its consistency was similar to Para gum rubber and it was practically impervious to water. The strength was taken as 100%. This expl is now known as *Blasting Gelatin* (qv) and the formulation comprising NG 98 & CC 8% is one of the Brit "standard" gelatinized explosives. Later formulations contd other ingredients, such as WM (woodmeal) or inorganic nitrates, known in the US as "dope". Such expls were softer than Blasting Gelatin because they contained less CC. Two of such gelatinized expls were Brit "standards", namely: *Gelatine Dynamite* – NG 75, CC 5, WM 5 & K nitrate 15% and *Gelignite* – NG 60, CC 4, WM 8 & K nitrate 28% (Ref 2). Such expls can be called "Straight Gelatins"

When a part of NG is replaced with NGc (Nitroglycol), the so-called LF (Low-Freezing) Gelatins are obtd. When part of NG (and of NGc) is replaced with AN (Ammonium Nitrate) in such quantities as to produce the same strength as before the replacement, the expl becomes *Extra LF Gelatin* (Ref 5)

The gelatinization of Nobel's Gelatins was facilitated either by warming to ca 60° or by adding volatile solvent, such as acetone, in cold. Later it was discovered that gelatinization is greatly facilitated not only by the addn of volatile solvents, but also by the addn of small quantities (sometimes as little as 0.1–1.0%) of substances, such as nitrated compds of benzene, toluene or xylene; urethanes, anilides, substituted ureas, etc. With these compds, gelatinization occurs in the cold. The soft gelatinous and semigelatinous

expls prepd in this manner, known as *plastic explosives* are used as "commercial explosives" and also as "military demolition explosives" (Refs 1 & 3)

The important discovery of Nobel revolutionized the mining expls industry because Gelatins were much more powerful than Guhr Dynamite, previously invented, and they practically did not exude. They were particularly suitable for blasting hard rocks

Stettbacher (Ref 4) prepd (1928–1929) a series of Gelatins consisting of PETN, NG and CollodCotton. These expls were called *Gelatine-Penthrinits* (See in this Vol) (Ref 4)

A series of new plastic expls based on RDX were prepd during WWII in the USA. They were designated as Composition C, C-2, C-3 & C-4 (See in Vol 3, pp C484-L to C486-L)

Refs: 1) Marshall 1 (1917), 364 2) Barnett (1919), 104 3) Marshall 3 (1932), 106 4) Stettbacher (1948), 86 5) J. Berliner, "Explosives", Pamphlet, NY (1953), 14 & 17 6) Cook (1958), 9 7) CondChemDict (1971), p 412 (Gelatin Dynamite)

Gelatin, 100%. DuPont & Co name for their Blasting Gelatin

Gelatina. Italian and Spanish name for Gelatin

Gelatina 808. Ital nonpermissible expl listed in Vol 3, p C440-L

Gelatina-dinamite N.O. Ital nonpermissible expl listed in Vol 3 of Encycl, p C440-L and in Giua, Trattato, VI(1),(1959), p 344

Gelatina esplosiva. Ital for Blasting Gelatin. Same as Gelatina gomma

Gelatina explosiva. Span for Blasting Gelatin

Gelatina explosiva al collodión. Span Gelatin consisting of NG & Collodion Cotton exploding accdg to the equation:

$51C_3H_5(NO_2)_3 + C_{24}H_{21}(NO_3)_9O_{11} = 177CO_2 + 143H_2O + 81N_2$

Ref: Vivas, Feigenspan & Ladreda, Vol 2 (1946), p 391

Gelatina explosiva al fulmicotón. Span Gelatin consisting of NG & Guncotton exploding accdg to the equation:

$41C_3H_5(NO_2)_3 + C_{24}H_{29}(NO_3)_{11}O_9 = 147CO_2 + 117H_2O + 67N_2$

Ref: Vivas, Feigenspan & Ladreda, Vol 2 (1946), p 391

Gelatina explosiva de guerra. Span name for the military expl invented in 1878 in Austria (See in Vol 5 under DYNAMITE, Class X, p D1610-L). It was used during Spanish-American War (1898) for loading projectiles fired from pneumatic cannons. Its compn: NG 86.40, NC 9.60 & camphor 4.00% was given by Vivas, Feigenspan & Ladreda, Vol 2, p 393. Russians used a similar expl contg 3% camphor for loading (during WWI) trench mortar shells, while Italians used the expl contg 5% of camphor (See Vol 5, p D1610-L)

Gelatina gomma. Ital for Blasting Gelatin contg NG 92 & NC 8%

Ref: Belgrano (1952), pp 85–6

Gélatine (Fr): NG 57, Collod Cotton 3, K or Na nitrate 34, sawdust 4, cereal flour 1.8 & red ocher (colorant) 0.2%

Ref: Pepin Lehalleur (1935), p 334

Gélatines (Belg & Fr). Daniel (Ref) gives compns of older Belgian and French Gélatines contg % NG: 86, 84, 83, 82, 74, 70, 69.5, 67, 50, 45, 43 and 25

Ref: Daniel (1902), pp 328–29

Gélatines, French (Low-Freezing). See Vol **5**, p D1591, Table I, under DYNAMITE

Gelatine A, Blasting (Brit): NG 63.4, Collod Cotton 1.6, K or Na nitrate 26.75, sawdust 8.40 & Ca carbonate 0.35%
Ref: Pepin Lehalleur (1935), p 335

Gelatine-Aldorfits. They are Swiss plastic expls contg NG, manufd by the Schweizerische Sprengstoff-Fabrik AG, Dottikon. Compn of one of them was: NG with NGc 22, Collodion Cotton 1.5, liq DNT with TNT 13 & AN 63.5%. Its props were: Trauzl Test value 400cc for 10g sample (vs 310 for TNT); Brisance (by copper cylinder compression) 3.25mm (vs 2.86mm for TNT); detonation velocity 6000m/sec at d 1.49 (vs 6970 for TNT at d 1.60); impact sensitivity with a 5kg weight 35cm (vs 45 for TNT); and explosion temp at the rate of 20° per minute 185°

There were also manufd Gelatine Aldorfits B & C and Gelatine Aldorfit with 20% Al
Refs: 1) A. Stettbacher, "Spreng- und Schiesstoffe", Rascher Verlag, Zürich (1948) 2) Dr A. Stettbacher, Private Communication, Zürich, Dec 14, 1953

Gélatine à l'ammoniaque A ou n° 2: A Belg explosif de sûreté: NG 30, Collod Cotton 3 & AN 67%
Ref: Gody (1907), pp 702 & 712

Gelatine-Astralit (Ger). A plastic low-freezing Dynamite contg DNClH with NG 30, DNT+TNT 10, AN+Na nitrate+woodmeal 60%. Its density 1.45; lead block expansion 400cc; lead block crushing 18mm; velocity of detonation 7300 m/sec; can be initiated by No 3 cap
Refs: 1) Naoúm, NG (1928), 379 2) PATR **2510** (1958, p Ger 66-L

Gelatine B, Blasting (Brit). NG 43.9, Collod Cotton 1.1, K or Na nitrate 41.25, sawdust 13.20 & Ca carbonate 0.35%
Ref: Pepin Lehalleur (1935), p 335

Gelatine, Blasting (Low-Freezing) (Ger & Swiss). See Vol **5**, p D1591, Table I, under DYNAMITE

Gelatin Blasting Explosive of J. Taylor et al, BritP 670453 (1952) & CA **46**, 10625 (1952): 80/20 mixt NG/NGc 57.5, NC 2.4, woodmeal 10.0, Na nitrate 24.8, finely milled barytes 5.0 & chalk 0.5%. Claimed to be suitable for work under a hydrostatic pressure of 40 atm. See also USP 2677605 (1954)

Another expl of J. Taylor et al, BritP 670454 (1952) & CA **46**, 10625 (1952), was claimed to be suitable for work in seismic prospecting and for well blasting. It consisted of an 80/20 mixt of NG/NGc 57.5, NC 2.5, peanut meal (deproteinized & de-oiled, ground) 13, Na nitrate 26.4, chalk 0.3 & diammoniumphosphate 0.3%

Gelatine-Carbonit (Ger). Several varieties exist as can be seen from Table G14

Table G14

Ingredients and some properties	Gelatin-Carbonites I	III	D	No designation
Am nitrate	31.0	46.4	31.5	41.5
Na nitrate	4.4	7.0	-	-
K nitrate	-	-	5.1	-
NG (mixed with collod cotton)	23.6	10.1	30.0	26.0
Glycerin plus gelatin	4.0	5.0	2.5	6.9
Na chloride	24.0	27.5	30.9	25.5
Vegetable meal	10.0	4.0	-	-
TNT	3.0	-	-	-
Ultramarine	-	-	-	0.1
Oxygen Balance,%	-13.1	+2.2	+5.3	-
Trauzl Test, cc	220	200	225	260
Vel of Detonation, m/sec	-	-	-	2300

Refs: 1) Naoúm, NG (1928), 407, 411 & 441 2) PATR **2510** (1958), p Ger 66-R

Gélatine-Carbonite, listed in Gody (Ref), contd: NG 27.0, NC 0.7, glue of gelatine-glycerin 6.5, AN 29.0, Na nitrate 22.8 & Amm chloride 14.0%
Ref: Gody (1907), p 713

Gelatine-Cheddit (Ger). Gelatinous expls based on chlorates, such as Na chlorate 70 & Collodion Cotton gelatinized with NG. They might serve as substitute Dynamites
Refs: 1) Naoúm, NG (1928), 353 2) PATR **2510** (1958), p Ger 66

Gelatine-Cheddite (Swiss). Plastic chlorate expls invented in 1911 by C. Rubins and then manufd by the Schweizerische Sprengstoff AG Cheddite and Dynamit. These expls are stable and do not harden in storage. The prepn and compn of one of the earliest formulations is given by Stettbacher in Ref 1. In Ref 2, p 87 the compn of newer *Gelatine-Cheddite* is given as follows: Na chlorate 79 (coated with a liq mixt of castor oil 5, liq DNT 2 & TNT 14 parts, preheated to ca 40°). Some Collodion Cotton could be incorporated. Its props are given in Ref 2 on p 104 as follows: max density 1.97, practical d 1.8–1.9; specific volume (vol of gases at NTP) 340 l/kg; heat of expln (at const vol, water vapor) 1100kcal/kg (2635kcal/l) ; temp of expln 3500°; max deton vel 3800m/sec; impact sensitivity with 2kg weight 50cm vs 90cm for TNT; and Trauzl test value 257cc for a 10g sample vs 310 for TNT at density 1.55
Refs: 1) A. Stettbacher, "Schiess- und Sprengstoffe", J.A. Barth, Leipzig (1933), p 313 2) A. Stettbacher, "Spreng- und Schiesstoffe", Rascher, Zurich (1948), pp 87, 91, 104 & 114 3) Dr A. Stettbacher, Private Communication, Zürich, Dec 14, 1953

Gelatine-Dahmenit (Ger). A type of low-freezing gelatinous Dynamite manufd for a long time by the Rummenohl Plant of the Westdeutsche Sprengstoffwerke of Dortmund. Two formulations are listed in Table G15

Table G15

Ingredients and some properties	Gelatin-Dahmenites A	B
Dinitroglycerin	27.4	27.4
Collodion cotton	0.6	0.6
Nitrotoluenes	4.5	3.5
Naphthalene	0.5	–
Ammonium nitrate	32.0	32.0
Potassium nitrate	2.0	2.0
Sodium nitrate	5.5	4.5
Alkali chloride	27.5	30.0
Trauzl Test, cc	233	205
Charge limit in firedamp, grams	350	700

Refs: 1) Naoúm, NG (1928), 419 2) PATR **2510**(1958), p Ger 66

Gelatin Detonating Explosive Containing Chopped Straw. Cereal straw cut to 1/3–1cm length and 0.3–1.3mm width, and having bulk density of 0.21 under 25psi does not impair the sensitivity or vel of a gelatin dynamite at the 9.5% level on storage at 35° as it has minimal absorption of the liq expl ingredient
Ref: C.R.S. Harries & J.C. Homel, BritP 578885(1946) & CA **41**, 1842(1947)

Gelatine Diaspon. See Diaspon Gelatine in Vol **5** of Encycl, p D1553-L

Gelatine-Donarit (Ger). A type of gelatinous industrial expl which contd DNClH+NGc 30, AN 50 & other ingredients 20% (Refs 1 & 2). Another gelatinous Donarit is described in Ref 2, p Ger 38. It contains NGc 22, Collod Cotton 1, AN 55, Na nitrate 10, TNT 5, DNT (liquid) 6, woodmeal 0.8 & dye (caput mortuum)

Note: DNClH = Dinitrochlorohydrin; NGc = Nitroglycol

Refs: 1) F. Weichelt, "Sprengtechnik", Marhold, Halle/Saale (1953), 37 & 375
2) PATR **2510**(1958), pp Ger 38 & Ger 66

Gelatin Dynamites (Amer). Compns of 20, 30, 40, 50, 60, 80 and 100% are listed on p D1600, Table V, under DYNAMITE in Vol **5** of Encycl

Gelatine Dynamites (Brit). Three "standard" gelatinized explosives: "Blasting Gelatin", "Gelatine Dynamite" and "Gelignite" are described here under GELATIN(Explosive) (Ref 1, p 104 & Ref 2, p 345)

When the supply of K nitrate became scarce during WWI it was replaced with Na nitrate in British "standard" Gelatine Dynamite and Gelignite (Ref 1, p 105). Giua (Ref 3) gives the following compn: NG 71, Collod Cotton 6, K nitrate 18 & woodflour 5% for one of the Brit Gelatine-Dynamites

Refs: 1) Barnett (1919), pp 104 & 105 2) Davis (1943), p 345 3) Giua, Trattato **6**(1) (1959), p 344

Gélatine-Dynamites (French). Accdg to Giua (Ref), they include:

Gélatine-Dynamite A: NG 64, Collod Cotton 3, Na nitrate 24 & woodflour 9%

Gélatine-Dynamite B-potasse: NG 57.5, Collod Cotton 2.5, K nitrate 32.0 & woodflour 8%

Gélatine-Dynamite C-soude: NG 57, Collod Cotton 3, Na nitrate 34 & woodflour 8%

Ref: Giua, Trattato **6**(1) (1959), p 344

Gelatine-Dynamits (Ger). A type of gelatinous explosive introduced by Alfred Nobel in 1875. The strongest of these, "Sprengelatine", contained NG 92 & Collod Cotton 8%. It is described as "Blasting Gelatin" in Vol **2** of Encycl, pp B211-R to B212-L. Other Gelatins prepd by Nobel contained ingredients (such as inorg nitrates, vege-

Table G16

Components and Some Properties	Gelatin-Dynamites								
	81%	80%	75%	No 1	No 2	No 3	No 4a	No 4b	No 5
NG	75.8	75	70.4	62.5	56–61	40	40	40	18–20
Collod Cotton	5.2	5	4.6	2.5	1–5	–	–	–	–
Vegetable meal	3.8	5	5.7	8.0	3–8	6	7	2	2–4
DNT + TNT	–	–	–	–	0–4	10	–	–	12
K nitrate	15.2	15	19.3	–	–	–	–	5	–
Na nitrate	–	–	–	27	–	44	41	–	–
Alkali nitrate and/or K perchlorate	–	–	–	–	25–30	–	–	41	54
Alkali chloride	–	–	–	–	–	–	12	12	12
OB to CO_2	–	–	–	+4.4%	–	+7.0%	+12.0%	+2.5%	+11.0%
Density	–	–	–	1.55	–	1.6	1.7	1.8	1.8
Lead Block Expansion	–	–	–	400	–	290	230	330	250
Lead Block Crushing	–	–	–	20	–	18	19	20	18
Max Velocity of Detonation	–	–	–	7000	–	6500	6500	6500	6500
Heat of Explosion kcal/kg (H_2O vapor)	–	–	–	1235	–	1030	850	1150	800
Temp of Explosion, °C	–	–	–	2950	–	2800	2500	3000	2650

table meal, etc) called "Zumischpulvem", known in the USA as "dopes"

Ger Gelatine-Dynamits may be subdivided into the following groups:
A. Gewöhnliches und Schwehrgefrierbares – ordinary and difficultly freezing (low-freezing)
B. Phlegmatisiertes, transportsicheres – phlegmatized, safe to transport
C. Schlagwetteres oder Wettersicheres – safe in the presence of firedamp(permissible)

To the A type belong the Blasting Gelatin and the Gelatins shown in Table G16

Any of these expls may be rendered low-freezing by incorporating NGc (Nitroglycol), DNG (Dinitroglycerin) or DNClH (Dinitrochlorohydrin). Aromatic nitrocompounds, such as DNT and TNT, can be used for the same purpose

To the B group belong Dynamites in which most of NG is replaced by DNClH, as for example, in Gelatine-Prosperits listed in Table G18, p G52

To the C group belong Dynamites contg small amts of NG and appreciable amts of "cooling" agents, such as alkali chlorides, as for example Gelatin-Dynamite No 4a, listed in Table G16 and Gelatine-Prosperit. Dynamites which contain large amts of AN (Ammonium Nitrate) also belong to this group (See Ammongelatine and Gelatine-Prosperit)
Refs: 1) Naoúm, NG (1928), 331, 334 & 349–50 2) Stettbacher (1948), p 85 3) PATR **2510**(1958), pp Ger 66 & Ger 67

Gelatine-Dynamits (Ger & Swiss). Formulations No 1, No 2 & No 3 are given on p D1601, Table VI, under DYNAMITE in Vol **5** of Encycl

Gelatin-Dynamite (Japanese). See under Permissible Explosives in Vol 3, p C454-L

Gelatin-Dynamite Containing Ammonium Nitrate. AN 50, urea 25, Na acetate trihydrate 15 & Na thiosulfate pentahydrate 10 parts are warmed until no solid remains; then 80 parts of this liq is mixed with 20 parts of NG containing 1% sol NC
Ref: J. Whetstone & J. Taylor, BritP 597716(1948) & CA **42**, 4349(1950)

Gelatin Dynamite Containing Ammonium Nitrate(Japanese). NG 28, NC 1.2, AN 43.3, starch 10, NaCl 20.0, soft wax 0.5 & urea 6% are pressed and wrapped in stick form.
Ref: K. Hino, JapP 2247(1953) & CA **48**, 4840(1954)

Gelatin-Dynamites of Apache Powder Co, Benson, Arizona. See Vol **5**, p D1602, Tables VII & VIII, under DYNAMITE

Gelatin Dynamite for Blasting Under High Liquid Pressure. Consists of blasting dynamite 97 (NG, NC, $NaNO_3$, wood pulp) and resin-treated corncob meal 3%. The latter is 25% heat-hardenable urea- or phenol-formaldehyde resin and is ground to 40 mesh after curing
Ref: C.D. Bitting & R.W. Lawrence, USP 2433417(1947) & CA **42**, 7046(1950)

Gelatin Dynamite Containing Chopped Straw. See Gelatin Detonating Explosive Containing Chopped Straw in this Vol

Gelatin-Dynamite Containing Crystalline Salts. An 80/20 mixt of NG/NGc 21 is used to dissolve P-NO_2Toluene 3 and p-tert-octyl-phenyl diethylphosphate 0.2 parts; gelled with NC 1.1, then mixed with AN 56.9, $NaNO_3$ 12, oat husk meal 2, waxed woodmeal 0.5, woodmeal 1, sulfur 2, starch 0.5 & chalk 0.3 parts
Ref: H.R. Wright et al, BritP713758(1954) & CA **49**, 3537(1955); USP 2716056(1955) & CA **49**, 15242(1955)

Gelatin-Dynamite Containing Glycol Ethers. Improved plasticity and ease of extrusion of

gel dynamites is obtained by incorporation 0.2–0.4% of a polyethylene glycol having at least 8 carbon atoms together and at least 2 ethyleneoxy groups together
Ref: S. Fordham & J.L. Moilliet, USP 2454643 (1948) & CA **43**, 3619 (1949); BritP 586224 (1947) & CA **41**, 7120 (1947)

Gelatin-Dynamite with Improved Plasticity. Incorporation of dried vegetable protein or protein-rich meals, such as soybean, into NG dynamites increases resistance to NG leakage due to sudden or large temperature chan ges
Refs: 1) M. Bonotto, USP 2413946 (1947) & CA **41**, 2246 (1947) 2) S. Fordham et al, BritP 586224 (1947) & CA **41**, 7120 (1947) (Inclusion of 0.4% of polyethylene glycol ether contg at least 8 carbon atoms)

Gelatin-Dynamites, Low-Freezing. American compns of 25, 40 and 60% LF Gelatin Dynamites are listed in Vol **5** of Encycl, p D1592, Table II, under DYNAMITE

Gelatine-Dynamits, Low Freezing. German LF Gelatine-Dynamits are listed in Vol **5**, p D1591, Table I under DYNAMITE

Gelatin-Dynamites Containing Nitroglycerin Fixed by Proteins. See Gelatin-Dynamite with Improved Plasticity

Gelatin-Dynamite Containing Oxazoline. Improvements in plasticity and extrudability of gel dynamites are made by including 0.01–0.1% of preferably, either 2-heptadecenyl-4-methyl-4-hydroxymethyl-2-oxazoline or 2-heptadecenyl-4-methyl-4-(hydroxydiethoxymethyl)-2-oxazoline
Ref: Atlas Powder Co, BritP 725438 (1955) & CA **49**, 14325 (1955); USP 2727814 (1955) & CA **50**, 4510 (1956)

Gelatine-Dynamite Containing Proteins. See Gelatin-Dynamite with Improved Plasticity

Gelatin-Dynamite Containing S-Hollow Pellet. Improved high water pressure performance of a gel dynamite is achieved by incorporating hollow pellets of sulfur coated with clay or wax
Ref: W.L. Reinhart, USP 2674526 (1954) & CA **48**, 7904 (1954)

Gelatin-Dynamite Containing Surface Active Agents. Triethanolamine oleate 0.1 is added to NG 21 and NC 0.5 at 120°F; then a blend of AN 14.1, Na nitrate 49.1, carbonaceous combustible material 9.0, sulfur 6 & chalk 0.3 parts is added
Ref: R.C. Glogau, USP 2676877 (1954) & CA **48**, 13222 (1954)

Gelatin-Dynamites, Straight and Extra. Accdg to one source, the following *straight* formulation: NG 50, NC 1.5, Na nitrate 38.0, carbonaceous matter 9.5 & chalk 1.0% is suitable for *tough* blasting. The formulation called *extra:* NG 28.5, NC 0.5, AN 30.0, Na nitrate 30.0, carbonaceous matter 10.0 & chalk 1.0% is suitable for *easy* blasting
Ref: Source, misplaced

Gelatin-Dynamite Containing Triethanolamine Salt. Into a 70/30 blend of NG/NGc 20, DNT 2 & NC 0.3 stir for 5 min triethanolamine oleate 0.025 and a mixture of C_{10-18} monohydric alcohols 0.025; then add AN 14, Na nitrate 49, carbonaceous material 7.9, sulfur 6.5 & chalk 0.3 parts
Ref: R.G. Glogau, USP 2676878 (1954) & CA **48**, 13223 (1954)

Gelatin-Dynamite Containing Water. Mix AN 57.1, K nitrate 5, carbonaceous fuel 7.8, NC gel 22.8, guar gel 8 (8–12 parts water per part guar). The result is a gel dynamite insensitive to a No 2 cap in the open, compared

to the No 1 cap sensitivity of a gel of the same sp gr and deton velocity but prepd with NC gel 30 and no guar gel
Ref: T. Sakurai, USP 2847291 (1958) & CA **52**, 19144(1958)

Gelatine-Explosives. They include compns listed here as Gelatins, Gelatine Aldorfit, Gelatine Astralit, Gelatin Dynamites, etc

Gelatine-Gamsit, also known as **Gamsit.** One of the Swiss, nonpermissible Gelatine Dynamites, manufd by the Société Suisse des Explosifs, Gamsen bei Brigg. The formulation of NG with NGc 22, Collod Cotton 1.5, DNT with TNT 13 & AN 63.5%, given by Stettbacher (Ref 2) on p 86, under "Schweizerische Nitrogelatinedynamiten" seems to be identical with that of *Gelatine Aldorfit* manufd by the Schweizerische Sprengstoff-Fabrik AG, Dottikon and with *Gelatine-Telsit*, manufd by the Schweiz Sprengstoffe AG at Isleten

Accdg to Naoúm (Ref 1) compn of Gamsit is the same as for Gelatine-Telsit (qv)
Refs: Naoúm, NG (1928), p 363 2) Stettbacher (1948), p 86

Gelatine-Leonit (Ger). One of the permissible, low-freezing Gelatine-Dynamits contg Dinitroglycerin, manufd before WWII by Rummenohl Fabrik of the Westdeutsche Sprengstoffwerke of Dortmund. Other Dynamites of the same type were Gelatine-Dahmenit and Gelatine-Tremonit
Refs: 1) Naoúm, NG (1928), p 418 2) PATR **2510**(1958), p Ger 67-L

Gelatine-Penthrinits and Penthrinits. The late Dr Alfred Stettbacher of Zürich, Switzerland invented in 1928 plastic, nonexudable, expl blends of PETN with NG to which later other ingredients were added. One of the first compns consisted of PETN 80 & NG 20% and was found to be suitable for use as a base chge in detonators with 0.04g LA as a primary chge. Another compn was PETN 85 & NG 15% (Ref 1). This expl was tested in 1933 by A. Izzo in Italy and found to be stable after storage for 28 days (quoted from Ref 4, p 223)

If the amt of PETN in formulation is below 60%, it is necessary to incorporate up to 6–7% CC (Collodion Cotton) in order to improve the plasticity in resulting blend. Such Dynamits became known as **Gelatine-Penthrinits.** If less brisant expls are desired, some AN (Ammonium Nitrate), up to 50%, may be incorporated, and such expls are known as **Ammon-Penthrinits.** Compositions and some properties of such expls are given in Vol 1, p A382-R, Table. For low-freezing Penthrinits, NG is mixed with NGc (Nitroglycol). Two formulations contg NGc are listed among Ammon-Penthrinits

Penthrinits and Gelatine-Penthrinits were prepd before WWII for research purposes by the Société Suisse des Explosifs, Gamsen bei Brigg and proved to be outstanding expls. Later they proved to be very effective for underwater explns. Two Penthrinits, 1 & 2 of Table 17 were, accdg to Stettbacher (Ref 6), loaded in 1929–1930 in 2cm shells and tested at Oerlikon Fabrik at muzzle velocity (v_o) 840m/sec. No prematures occurred. The addition of about 15% Al powder increases the efficiencies of Penthrinits, while high amounts seem to decrease it. For example, addition of 30% Al to formulation 1 of Gelatine-Penthrinits decreased detonation velocity from 8000m/sec to 7200

The Gelatine-Penthrinits 1, 2 & 3 were listed in Table 17, Vol 3 of Encycl, p C443-R, but their properties were not given. In the same place compn 4 was listed under the name Gelatine-Penthrinite-Perchlorite, but its properties were not given

Six Penthrinits and four Gelatine-Penthrinits formulations are given in Table G17 including some properties

Table G17
Penthrinits and Gelatine-Penthrinits of Alfred Stettbacher

Composition & Properties	Penthrinits						Gelatine Penthrinits			
	1	2	3	4	5	6	1	2	3	4
PETN	80	85	70	50	40	80	50	59.0	15.5	50
NG	20	15	30	50	60	15	46	24.7	77.5	46
NGc	–	–	–	–	–	5	–	–	–	–
CC	–	–	–	–	–	–	4	0.6	7.0	4
NH_4ClO_4	–	–	–	–	–	–	–	15.7	–	–
Al (added)	–	–	–	–	–	–	–	–	–	30
Density (loading	1.49	–	1.50	–	–	–	1.57	–	–	1.85
Density (max)	1.72	–	1.71	1.68	1.67	1.71	1.68	–	–	1.96
Detonation Velocity,m/sec	8600	–	8500	8400	8200	8600	8000	–	–	7200
Trauzl Test, cc	530	–	510	–	–	528	–	–	–	–
Pb Block Compression,mm	–	–	29	–	–	–	–	–	–	–
Impact Test, 2kg Wt, cm	2.9	–	–	–	–	–	17	–	–	–
Heat of Expln (H_2O vapor), kcal/kg	1450	1438	1473	1519	1542	1447	1549	–	–	2000
Volume of Gases at NTP in liters/kg	762	766	–	–	–	–	744	–	–	520
Temperature of Expln,°C	–	–	–	–	–	–	4750	–	–	

Refs: 1) A. Stettbacher, SS **23**, 345–48 (1923) 2) Ibid, AngewChem **43**, 844–47 (1930) 3) A. Stettbacher, Nitrozellulose **4**, 222–27 (1933) 4) Ibid **5**, 6–12 (1934) 5) A. Stettbacher, "Spreng- und Schiesstoffe", Rascher, Zürich (1948), 83–5 & 104 6) Dr A. Stettbacher, Private Communication, Zürich, Dec 14, 1953

Gelatine-Prosperit. (Ger). A type of low-freezing, gelatinous Dynamite based on (DNClH) Dinitrochlorohydrin. Table G18 lists two formulations:

Table G18

Components and Some Properties	Gelatine-Prosperit	
	No 1	No 2
DNClH	20.0	20.0
NG	5.0	5.0
Collod Cotton	0.5	0.5
DNT	5.0	5.0
Cereal meal	2.5	2.0
Amm nitrate	36.0	30.0
Na nitrate	4.0	10.0
Na chloride	21.0	27.0
K oxalate	6.0	–
OB to CO_2	0.4%	–1.2%
Trauzl Test, cc	225	210

Compare with Gelatine Wetter-Astralit

Refs: 1) Naoúm, NG (1928), 418 2) PATR **2510**(1958), pp Ger 67 & Ger 68

Gelatine-Romperit (Ger). Accdg to F. Weichhelt, "Sprengtechnik", C. Marhold, Halle-Saale (1953), p 37, its compn is the same as that for Gelatine-Donarit

Gelatine-Telsit (Swiss). An expl manufd by Schweizerische Sprengstoffe AG Cheddite & Dynamit at Isleten for blasting in construction of tunnels thru the Alps. Its compn was, accdg to Naoúm (Ref 1), NG 22.0, NC 1.5, DNT 21.0 & AN 55.5% and properties: OB to CO_2 –12.6, density 1.46, Trauzl test value 380cc, lead block compression 14.6mm and deton velocity 6700m/sec. Could be initiated with a No 6 Blasting Cap

The same compn is given by Stettbacker (Ref 2) for *Telsit* with the same props as above, except density is given as 1.55. The same author gives in his later book (Ref 3) for *Gelatine-Telsit, Gelatine- Gamsit* and *Gelatine-Aldorfit* the following compn: NG (with or w/o NGc) 22.0, Collodion Cotton 1.5, DNT with TNT (called flüssiges Tri) 13.0 & AN 63.5%. Its props are given under Gelatine-Aldorfit

Refs: 1) Naoúm, NG (1928), p 363
2) Stettbacher (1933), p 242 3) Stettbacher (1948), p 86

Gelatine-Tremonits (Ger). Gelatinous, low-freezing mining expls manufd before WWII by the Castroper Sicherheitssprengstoffe at Hagen, Westfalia

Two of the formulations are shown in Table G19

Table G19

Ingredients and Some Properties	Gelatine-Tremonits	
	I	II
Gelatinized Di-Trinitroglycerin mixture	47.5	30.0
DNT	5.0	10.0
Woodmeal	5.0	2.0
Amm nitrate	22.5	40.0
Na nitrate	20.0	18.0
Trauzl Test, cc	400	375

Refs: 1) Naoúm, NG(1928), 368 2) PATR **2510**(1958), p Ger 68

Gelatines, Vergé (Swiss & Ger). Accdg to Naoúm (Ref 2), Vergé received in 1913 Swiss patents for the four expls, listed here in Table G20

Table G20

Composition & Properties	Gelatine 62%	Gelatine 56.5%	I	II
Nitroglycerin	40.0	37.0	38	41
Liquid Nitrocompounds	17.0	15.0	16	18
Guncotton	5.0	4.5	–	–
Collodion Cotton	–	–	2	3
NH_4ClO_4	38.0	25.2	26	–
$KClO_4$	–	–	–	38
$NaNO_3$	–	18.3	18	–
OB to CO_2	–5.4%	+1.1%	+1.0%	–1.1%
Trauzl Test	–	–	450cc	405cc
Pb Block Compression	–	–	20.6mm	18.21mm
Sensitiveness	–	–	Cap No 1	Cap No 1
Detonation Velocity, m/sec	–	–	6600	7300
Density	–	–	1.52	1.57

Note: Liquid nitrocompounds consisted of TNT 20, dissolved in liq m-DNT 80
Refs: 1) A.E. Vergé, SS **10**, 104 & 158(1915)
2) Naoúm, NG(1928), pp 364 & 365
3) Vol **5** of Encycl, p D1591, Table 1, under DYNAMITE

Gelatine-Wetter-Astralit (Ger). Compositions of two mining expls based on DNClH (Dinitrochlorohydrin) are, accdg to Naoúm (Ref 1), identical with *Gelatine-Prosperits* described here

A different compn, however, is given in Thorpe's (Ref 2): CNClH 16.0, NG 4.0, Collod Cotton 0.5, MNT 1.0, DNT 4.0, AN 40.0, flour or potato meal 8.0, Na nitrate 7.5, charcoal 0.5, castor oil 2.0, Amm oxalate 2.5 & NaCl 14.0
Refs: 1) Naoum, NG(1928), p 418
2) Thorpe Dict, Vol **4**(1940), p 554

Gelatine-Wetter-Nobelit (Ger). A permissible, gelatinous expl: NG (gelatinized) 30.0, AN 26.5, woodmeal 0.5, NaCl 40 & 3% of Ca nitrate in 50% aqueous soln
Refs: 1) Stettbacher (1948), pp 91–2
2) PATR **2510**(1958), p Ger 68

Gelatinization (Gelation or Jelling). It is conversion of a substance into a gel, such as by treating NC with NG or other solvents, such as acetone or mixture of ether-ethanol. Gelatinization of NG with NC is described under GELATIN in this Vol

Gelatinization of NG with NC in prepn of smokeless propellants is described in Vol **3**, pp C398-L to C403-L, under "Colloiding Agents and Colloidal Propellants"

Gelatinization Accelerants. In gelatinizing NC with a liquid expl, such as NG, the addition of 0.5–2% of a derivative of an aliphatic alc contg at least 2 hydroxyl groups, such as ethyleneglycol monoethyl ether, facilitates the process. Such substances as EGcMEE may be called "gelatinization accelerants"
Ref: H.H. Fassnacht, USP 1966090(1934) & CA **28**, 5672(1954)

Gelatinization Coefficient. See Coefficient de gélatinisation or Pouvoir gélatinisant (Fr) in Vol **3** of Encycl, p C389-L & R

Gelatinization or Jelling Coefficient of Soler(G). Under the term *coeficiente de gelatinizacion*, Soler & Vian (Ref 1) and Soler (Refs 2 & 3), designated G as weight in grams of NC of nitrogen content 11.80% or of 13.0% which became completely gelatinized after being kept in contact for 2 hours with 1 g of gelatinizer to be tested. It was assumed that 2 hours of contact gives sufficient time for gelatinization if there is any

In testing gelatinization of 13% N NC, three substances were examined: diphenylurethane, urethane and Et Centralite. In general, gelatinization increased with the temperature and at lower temps G's were different with each gelatinizer. It became, however, equal for all 3 substances at 174°. The work was extended later to MNPhenol; o-MNT; 2,4-DNT; tritolylphosphate and Et oxalate

An equation was derived for calculating G, in advance for various temps
Refs: 1) A. Soler & A. Vian, Ion(Madrid) **2**, 745–52 & 833(1942) 2) A. Soler, Anales de la Universidad de Murcia, **1943**, p 25
3) A. Soler, Anales de Fisica y Quimica (Madrid) **40**, 266–80(1944)

Gelatinization Coefficient of Nitrocelluloses and Coefficient de plasticité. In the paper entitled "Plasticités des pâtes de Poudre B", the term "coefficient de plasticité" is defined and a simple apparatus for its determination is described
Ref: Anon, MP **19**, 146–53(1922)

Gelatinization Rate for NC. The pulped & dried NC of various N contents was shaken and stirred with a mixture of 80/20 NG/NGc. Using a standard electrophotometer the light absorption was read at 60-second intervals. It was concluded that the rate varied with a number of factors, including N content, viscosity, the state of pulping of fibers, etc
Ref: D. Fensom, CanadJRes **26B**, 59–69 (1948) & CA **42**, 4347 (1948)

Gelatinization of Various Nitrocelluloses Conducted at Picatinny Arsenal Before, During and After WWII. The actions of various solvents on the swelling, gelatinization, solubility, and plasticization of NC is described in Vol **2** of Encycl, pp C115-R to C118-R. A list of PA repts relating to this subject is found in the Refs below
Refs: 1) A.J. Phillips, "Study of the Efficiency of Various Agents in Gelatinizing Nitrocellulose", PATR **72** (June 1931) 2) Ibid, PATR **279** (Sept 1932) 3) J.D. Hopper, "Study New Solvents as Possible Ingredients of Smokeless Powder", PATR **952** (Feb 1939) 4) D.D. Sager, "Devlopment of a High Velocity Caliber .50 Powder", PATR **1023** (Jan 1940) 5) A.J. Philips, "Study of Factors which Affect the Volatility of Smokeless Powder", PATR **1024** (Jan 1940) 6) H. Aaronson, "Study of Explosives Derived from Nitroparaffins", PATR **1125** (Oct 1941) 7) A.J. Phillips, "Study of the Gelatinizing Action of Esters of Lauric, Phthalic, and Adipic Acids upon Nitrocellulose", PATR **1201** (Oct 1942) 8) A.J. Phillips, "The Relation of the Hydrogen Bonding Concept to the Gelatinization of Nitrocellulose", PATR **1249** (March 1943) 9) Ibid, PATR **1587** (Jan 1946) 10) B.T. Fedoroff, "Basic Research Leading to the Development of Ideal Propellants. Preparation of Low Viscosity, High Nitrogen Nitrocellulose", PATR **1678** (Feb 1948) 11) B.T. Fedoroff, "Basic Research Leading to the Development of Ideal Propellants. Non-Explosive Plasticizers for Nitrocellulose", PATR **1753** (Nov 1949) 12) T.C. Castorina, Ibid, "Explosive Plasticizers for Nitrocellulose", PATR **1755** (Dec 1949) 13) H.A. Aaronson, "Basic Research on Rocket Propellants High Polymer Systems. The Kinetics of Solution of Cellulose Nitrates in Explosive Plasticizers", PATR **1786** (Oct 1950)

Gelatinized Dinitrotoluene Explosives. J. Rudloff and W. Allendorf patented in 1906 in Germany expls which contained DNT previously gelatinized by mixing with 0.6% Collodion Cotton: eg, Gelatinized DNT 18, TNT 14, $KClO_3$ 60–68, & $Pb(NO_3)_2$ 0 to 8%. These expls were more powerful than straight TNT because they contained sufficient oxygen for complete combustion
Ref: Colver (1918), 678–79

Gelatinized or Gelatinous Dynamites. Their definition is given here under GELATIN, and various expls are described under the titles of Gelatine Astralit, Gelatine Carbonit, Gelatin Dynamite, Gelatin Dynamite etc

See also under DYNAMITE in Vol **5**, pp D1599 to D1602, Tables IV, V, VI, VII & VIII, and under GERMAN DYNAMITS AND THEIR SUBSTITUTES

Gelatinized or Gelatinous Explosives, French. See under French Gelatinized Explosives, which include Gommes, Gélatinée 1B, Gélignites, Dynamite gélatinée, etc, p F195-L

Gelatinized or Gelatinous Explosives, German. They are not segregated but described as individual expls, such as Gelatine Astralit, Gelatine Dahmenite, etc. A general description of German Dynamits (which includes Gelatine Dynamits) is given here under GERMAN DYNAMITS AND THEIR SUBSTITUTES and in Vol **5**, p D1599 to D1602, under DYNAMITE

Gelatinized Trinitrotoluene Explosives. Plastic, low-freezing expls, introduced in 1911 by the "Société Universelle d'Explosifs" of Paris, were prepd by mixing thoroughly at 40–50°, 21–24 parts of liquid TNT with 1 or 2 parts of Collodion Cotton and gradually adding 78–75 parts of K Chlorate

S. Nauckhoff patented the following mixture in 1911: Gelatinized TNT (consisting of 96% of liq TNT and 4% of mixt of Collodion Cotton 31, NH_4ClO_4 43, NH_4NO_3 26)

Ref: Colver (1918), 247

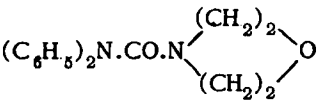
Gelatinizers and Stabilizers for Smokeless Propellants. Giua & Guastalla (Ref) listed the following compds proposed by them: aniline, Centralite, DPhA (Diphenylamine), phthalide, α-naphthylurethane, ethyl oxanilate ($C_6H_5.NH.COOC_2H_5$) and potassium oxanilate ($C_6H_5.NH.COOK$)

Ref: M. Giua & G. Guastalla, Chimie & Ind (Paris) **29**, 268–77 (1933) & CA **27**, 2580(1933)

Gelatinizers and Stabilizers for Smokeless Propellants. Lévy (Ref) described the prepn of the following substances which he called "nouveaux stabilisants et gélatinisants":

Diphenyl carbamyl piperidine $(C_6H_5)_2$-N.CO.N$(CH_2)_5$, crysts (from alc), mp 123.5°

Diphenyl carbamyl morpholine,



crysts (from alc), mp 108.5°

α-Naphthyl diphenylurea, $(C_6H_5)_2N.CO.NH.C_{10}H_7$, crysts (from alc), mp 185.5°

β-Naphthyl diphenylurea, same formula as above; crysts (from alc), mp 182°

Diphenyl carbamyl diethanolamine, $(C_6H_5)_2N.CO.N(CH_2CH_2OH)_2$, crysts (from alc), mp 124.5°

Diphenyl carbamyl monoethanolamine, $(C_6H_5)_2N.CO.NH.CH_2.CH_2OH$, crysts (from alc), mp 117.5°

Diphenyl carbamyl allylamine, $(C_6H_5)_2N.CO.NH.CH_2.CH{:}CH_2$, crysts (from alc), mp 82°

bis (Diphenylcarbamyl)-ethylenediamine, $(C_6H_5)_2N.CO.NH.CH_2.CH_2.NH.CON(C_6H_5)_2$, crysts (from alc), mp 184°

Ref: R. Lévy, MP **32**, 309–12(1950)

Gelatinizing Agents. Same as Colloiding Agents described in Vol **3**, pp C398-L to C403-L

Gelatinizing (or Plasticizing) Power of Substances in Explosives or Propellants. It is the ability of a substance, such as NC, PETN, Centralites, etc to bond a liquid expl (such as NG or NGc) or a nonexpl solvent (such as acetone or ether/alc) to a homogeneous or nearly homogeneous gelatinous or semigelatinous mass

For testing gelatinizing power of CC (Collodion Cotton) a special test was designed by Escales. The test as later modified and described by Naoúm (Ref) was conducted as follows:

A sample of 2.5g of finely screened CC was placed in a small porcelain casserole, stirred (using a hardwood, plastic or horn spatula) with 97.5g of cold NG. The casserole was then placed on a water bath, heated to 65° and the mixture kneaded for 20 minutes, while warm, with the same spatula. After this, the mass was cooled to RT without stirring and examined by cutting with a spatula. If the mass were cut in coherent lumps, which easily separated from the porcelain casserole, the gelatinizing power was called 1. If the mass were not very coherent and stuck slightly to the porcelain, its power was 2, and so on to the grade mark 5, depending on coherence and stickiness. CC which gave a very sticky or oily, non-cohesive gelatin was rejected because it would produce an easily-exudable Dynamite

Although this method was crude, it gave, with a little practice, fairly reliable results

Ref: Naoúm, NG (1928), 295

Gelatinous Dynamites with Explosive Base. See Vol 5 of Encycl, pp D1599 to D1602, under DYNAMITE

Gelatinous Nitrate Containing Polyisobutylene-Gelled Naphthene Base. Increased plasticity of dynamite-type expls is achieved by adding 1–10% of a gel based upon naphthene oil 95 and polyisobutylene 5 parts. Lubricating oil, heat-exchange oil or petrolatum may be substituted for the naphthene
Ref: J.M. Downard, USP 2537039 (1951) & CA **45**, 2670 (1951)

Gelatinous Permissible Explosive. One of the French compns was given by Naoúm (Ref): Dinitroglycerin 35.0, NC 0.7, AN 38.4, TNT 2.5, cellulose 2.0, Amm chloride 9.4 & Na oxalate 12.0%
Ref: Naoúm, NG (1928), 420

Gelation or Jelling. Same as Gelatinization

Gelatit I. A German permissible expl: NG (gelatinized by Collodion Cotton) 30.0, AN 35.0 to 37.0, woodmeal 0.5–1.5, DNT (or DNT with TNT) 0 to 2.0, KCl or NaCl 32.0 & coloring matter 0 to 5%
Refs: 1) Pepin Lehalleur (1935), 414–15 2) Beyling & Drekopf (1936), 100 3) PATR **2510** (1958), p Ger 68-L

Gelbin. One of the names for Calcium Chromate listed on p C279 of Vol 3 of Encycl

Gelbite of Emmens (Amer). See Vol 5, p E87-L, under Emmensite

Gelbpulver (Yellow Powder). A Ger expl of WWII: TNT 54, NGc 18, NC (of 12% N) 27 and stabilizer (Acardite or Centralite) 1%; colored yellow. Prepn and props are in Ref
Ref: Anon, German Report Rak 3, II, March 1943, translated at PicArsn Library by Dr G. Loehr

"Gelex". Trademark of DuPont & Co, registered in US Patent Office (Ref 3), for Semigelatin Dynamites (Nos 1 to 5 incl) having plasticity and water-resistance ratings between Ammonium Dynamite and Special Gelatin. Have a very good fume rating. Used in mining metallic ores, gypsum, limestone, in quarrying hard rock, and in construction work (Refs 1 & 2). Compositions are not given, but their bulk strengths, densities and detonation velocities are listed in Ref 3
Refs: 1) Cook (1958), 12 2) CondChemDict (1961), 530-L 3) Blasters' Hdb (1966), p 39, Table 4-3 and pp 40–1 & 510
4) PATR **2700**, Vol 5 (1972), p D1604-R, under DYNAMITE, Class VI

Gelgas or Gelled Gasoline. See under Flame Throwers-Liquids and Gels in Vol 6, pp F56ff

GELIGNITE (Gelignit, in Ger; Gélignite in Fr; Gelignite in Ital, Gelignita in Span; Ghelignit in Russ)

Accdg to Daniel (Ref 1), Gelignites are certain varieties of Gelatin-Dynamites, which originated in England at the end of the 19th century. He lists eight varieties authorized between 1897 & 1898 for use in coal mines contg firedamp (grisou) and dust (poussière). The names of these Gelignites were British, Kynox, Nahusen, National, Nobel, Rhenish, Stowmarket and Sun. Their compns range is NG 54 to 64, NC 2 to 6, saltpeter 24 to 34 & wood flour 5 to 10

Accdg to Marshall (Ref 2, p 369), Gelignite was introduced to replace Blasting Gelatine which was too violent for certain kinds of work, too local in its action and too expensive on account of large proportion of NG. Gelignites used during WWI consisted

of 56 to 63% NG thickened with CC (Collodion Cotton) to a thin jelly and mixed with K nitrate and WM (woodmeal), with the addition, sometimes, of Ca carbonate and mineral jelly. A typical compn for Brit Gelignite as given on p 370 of Ref 2 was: NG 60.5, CC 4.5, K nitrate 27.0, WM 7.0, Ca carbonate 0.2 & moisture 0.2%. For comparison, the compn of Brit Gelatine Dynamite used at that time was: NG 74.5, CC 5.5, K nitrate 15.5, WM 4.0, Ca carbonate 0.2 & moisture 0.3% and of Blasting Gelatine: NG 91.5, CC 8.0, Ca carbonate 0.2 & moisture 0.3%

As was mentioned here under GELATIN, the Gelignite contg NG 60, CC 4, K nitrate 28 & WM 8% is one of the Brit "standard" gelatinized expls (Ref 3, p 104) and its ballistic pendulum swing is 3.27 inches for 4 oz sample (Ref 3, p 184). Its deton velocity is 6600m/sec vs 7000m/sec for the above 74.5% Gelatine Dynamite (Ref 3, p 191). Another Brit Gelignite contd NG 63.5, CC 1.5, Na nitrate 27.0 & WM 8.0%, and its deton vel was 6210m/sec at d 1.67 (Ref 3, p 118)

Accdg to definition given by Taylor & Gay (Ref 7), Gelignites are industrial expls contg less NG–NC jelly and more woodmeal and O–contg salts than Gelatine-Dynamites. They are plastic in nature and should not be confused with "semi-gelatins" which are betw powdery and plastic expls. The Dynamites which are powdery are now called in GtBritain "Powders" (These seem to correspond with Amer Straight Dynamites)

McAdam & Westwater(Ref 8) list the following Brit Gelignites: 1) *Gelignite 62%.* NG+NC 65, Na nitrate 28 and combustible materials with stabilizer 7%; density 1.5, deton velocity 2500m/sec and temp of expln 3730°C

2) *Gelignite 42%.* NG+NC 45, Na nitrate 44 and combustible material with stabilizer 11%; density 1.55, deton vel 2500m/sec and temp of expln 3290°C

3) *Polar Ammon-Gelignite.* NG+NC 30, AN 58 and combustibles with stabilizers 12%; density 1.45, deton vel 2500m/sec and temp of expln 2685°C and for comparison:

Polar Ammon-Gelatine-Dynamite. NG+NC 35, AN 60 and combustibles with stabilizer 5%; density 1.40, deton vel 2500m/sec and temp of expln 3525°C

For comparison we are listing here the German *Gelignit II.* NG 47.5, Collod Cotton 2.5, K nitrate 37.5, woodmeal 3.5 & rye flour 9.0% (Refs 4, 5 & 9)

Gelignites, although originally industrial expls, were used during WWII by the British and American Armed Forces mostly for demolition charges, but also in some A/T mines and in Stickey Grenades. They were used under the names of *Saxonite* or *Nobel's 803(808, 223* or *853).* A typical compn was NG (gelatinized with 2–7% Collod Cotton) 50, the rest being a mixt of Na nitrate & woodmeal (Ref 6)

Accdg to Ref 10, commercial *Gelignite "60"* was used in demolition charges weighing 4 or 1½ oz, wrapped in wax paper. Gelignites "60" and "50" were used in Australian *Demolition Cartridges* weighing 5½ oz

Gelignites also were used in Belgium, France, Germany and Italy. In Spain it was not listed as Gelignita (See Ref 6)

Refs: 1) Daniel (1902), 339–40 2) Marshall 1(1917), 369–70 3) Barnett (1919), 104, 118, 184 & 191 4) Naoúm, NG(1928), 330 5) Davis (1943), 345 6) All&EnExpls (1946), 151–52 6) Vivas, Feigenspan & Ladreda, Vol 2(1946), 398 (Compn listed as *Dinamita goma núm 2:* NG 49.0, NC 2.5, Na nitrate 35.5 & flour 13.0% of Fábrica de Galdácano is similar to Brit & Ger Gelignites) 7) Taylor & Gay (1958), 24–5 8) McAdam & Westwater (1958), 29–31 9) PATR **2510** (1958), p 68-R 10) Anon, "Explosives and Demolitions", US Dept of Army Field Manual **FM 5–25** (1967), 10

Gélignite à l'ammoniaque. A Belgian permissible expl contg NG 29.3, AN 70.0 & CC 0.7%. It is identical with French *Grisoutine-gomme*

Ref: Daniel (1902), p 23

Gelignite, Ammon. See Brit nonpermitted expl listed as *Ammon-Gelignite* on p A368 of Vol 1 of Encycl, Table

Celignite all'ammonio. Ital nonpermissible expl listed in Vol 3 of Encycl, p C440-R

Gélignite d'Arendonck. Belg mining expl: NG 56.20, Collod Cotton 1.80, Na nitrate 31.29, woodflour 10.08 & Na carbonate 0.63%
Ref: Gody (1907), 360

Gélignites (Belg). Mining expls: 1) NG 58.6, Collod Cotton 2.5, woodpulp 8.0 & Na nitrate 31.0%; 2) NG 60.51, Collod Cotton 4.88, woodpulp 7.18 & K nitrate 27.48%
Ref: Gody (1907), p 375

Gélignites (Belg & Fr). Daniel (Ref) gives compns of Belgian and French Gélignites contg the following % NG: 57, 57.5, 57.86, 60 & 64. Other ingredients were NC, K or Na nitrate & woodmeal
Ref: Daniel (1902), pp 328–29, Table

Gelignite (Ital). Nonpermissible expl listed in Vol 3, p C440-L (bottom)

Gel for Incendiary Bombs. Addition of 8–15% lanosterol to petroleum hydrocarbons forms a solid gel suitable for use in incendiary bombs (See also Napalm)
Ref: P. Vaterrodt, USP 2719782(1955) & CA **50**, 3764(1956)

Gel-lacquer Application. Gel-lacquers are solns of polymer in relatively weak solvents, the compn of the soln being adjusted so that it is fluid at about 50°C. As the coated article is withdrawn from the bath, the air at RT cools the coating and immediately sets it to a nonflowing gel before sagging & running can take place. The lacquer subsequently goes thru the final drying stages with loss of solv in the usual time. If the article being dipped is cool enough or has sufficient heat capacity & good thermal stability, the layer of gel will form while the article is still immersed in the bath, and in this case an even thicker layer is applied. It is under these circumstances that dried layers of lacquer as thick as 0.015 inch can be applied

The solvents which have been employed are the cheaper paint & lacquer solvs. The gel-lacquer technique had been worked out for two classes of polymers, and in time may now be extended to most lacquer polymers
Ref: Dr L. Gilman, "Plastics Developments", PA Technical Div Lecture, Picatinny Arsenal, delivered 31 Jan 1947, pp 8–9

Gelled Gasoline. See Gelgas under Flame Throwers-Liquids and Gels in Vol 6, p F56ff

Gelled High Energy Oxidizers are described by J.W. Dale et al in confidential Quarterly Technical Report **No 1**, 25 Jan 1963 to 24 Apr 1963 and 25 May 1963 of Monsanto Research Corp, Contract N600(19) 59719

Gelled Liquid Nitroparaffins, such ad Nitromethane, contg 5–75% NG, some amine and up to 3% of Cr acetylacetonate, may be used as an expl or a propellant. The gels are liquid if small amts of NC are used, and solid if the amts are high
Refs: H. Maisner, USP 2690964(1954) & CA **49**, 618(1955)

Gelled Propellant Safety Investigation. Intermixing of gelled N_2O_4 & UDMH (unsymdimethylhydrazine) results in a fire if large surface areas of gelled fuel & oxidizer come in contact. The fire is similar to that which occurs when two hypergolic proplnts are

mixed in their liq state. A small controllable fire will occur when small surface areas of the gel come in contact

A large scale spill of liq proplnt will result in splashing & spreading over a large area. A spill of the same amt of gel will remain in a much smaller area and result in increased safety to personnel in the area
Ref: J.G. McCroskey, "Gelled Propellant Safety Investigation", Air Force Systems Command, Research and Technology Div, **RDT-TR-63-1069**, July 1963

Gelling (or Jelling) Point. The setting point (concentration and temperature) at which semiliquids or pastes become solid
Ref: Hackh's Dict (1944), 372-R

Gelling Normally Liquid Hydrocarbons. Benzene, 50 parts may be gelled by adding potassium tridecylxanthate 3 parts; naphtha 50 parts will further stabilize the gel. Possible uses are solid fuels, lubricants, military incendiaries & hydraulic fracturing fluids. Addition of w, 3–5%, will liquefy the gel
Ref: R.M. Alm, USP 2668098 (1954) & CA **48**, 5486 (1954)

"Gelobel". A series of DuPont gelatinous and semigelatinous permissible dynamites. Of these "Gelobel" AA has deton vel 16500ft/sec, when fired unconfined with Straight Dynamite primer. It has excellent water resistance and is intended for use in the most difficult types of rock work. The "Gelobel" C has deton vel 12100ft/sec, and good water resistance. It is more economical than AA and best suited to soft rock and jobs that do not require heavy burden. No compns of these expls are given in
Refs: 1) DuPont Blasters' Hdb (1966), pp 44 & 46 2) CondChemDict (1961), 530-L; (1971), not found

Gelose. Accdg to Hackh's (Ref 2), it is the same as *galactan* (qv), but accdg to Pérez Ara (Ref 3), it is the same as agar-agar. Under the name of "gelose", or "agar-agar", it was used as a plasticity controlling component of some expls, such as *Grenée* (Poudre). It is called in Ref 3 Pólvora Grenée. An older French expl contd NC 60, Ba nitrate 30, K nitrate 6, "gelose" (agar-agar) 3 & paraffin 1%. Naoúm (Ref 1) lists the compn of *Wetter-Wasagit A*, "gelose" 1%, the rest being NG (gelatinized) 28.0, AN 20.0, Ba nitrate 10.0, KCl 40.5 & talc 0.5%. It was manufd by the Westfälisch-Anhaltische Sprengstoff AG at Sythen and Reinsdorf Plants
Refs: 1) Naoúm, NG (1928), 415 (Wetter-Wasagit A) 2) Hackh's Dict (1944), 364 3) Pérez Ara, "Tratado de Explosivos", "Cultural SA", La Habana (Cuba) (1945), p 440

Gélosine. A substance contg about 98% water, obtd from some sea algae. It was recommended by Chalon & Guérin for use as tamping in boreholes of gaseous coal mines. It was claimed that vapor resulting from evaporation of algae water during expln, diminished the temp of gases of expln, thus preventing the danger of firedamp expln
Ref: Daniel (1902), 340

Geloxites. Brit safety explosives contg NG 54–64, K nitrate 13–22, Amm oxalate 12–15, woodmeal 4–7 & red ochre 1%
Ref: Clift & Fedoroff, Vol 2, p G2

Gelsemine, $C_{20}H_{22}N_2O$, mw 366.22; prisms (from acet), mp 178°; insol in w; sol in alc, ether, chlf, and dilute acids. A poisonous alkaloid obtd from rhizome and root of *Gelsemium sempervirens*. Used in medicine as an analgesic and antispasmodic
Refs: 1) Beil **27**, [720] 2) T.G. Wormley, AmJ Pharm **42**, 1 (1870) 3) Hackh's (1944), 372-R 4) CondChemDict (1961), 530-L & R; (1971), 412-R

Gelsemine, Nitration. Nitration of gelsemine yields only an amorphous material. Nitration of dihydrogelsemine in mixed acid at −7 to +5° gives **Dinitrogelsemine**, analyzing for $C_{20}H_{22}N_4O_6$, mw 458.22, yel ndls (chlf or alc), mp 257–8°; sol in chlf, pyridine; sl sol in acet, methanol, alc, benz, eth & w. The dinitro compd forms a **nitrate salt**, yel rhombic prisms, mp 219–21° dec
Ref: T.Q. Chou & T.T. Chu, JACS **63**, 827 (1941) & CA **35**, 2897 (1941) (Nitration)

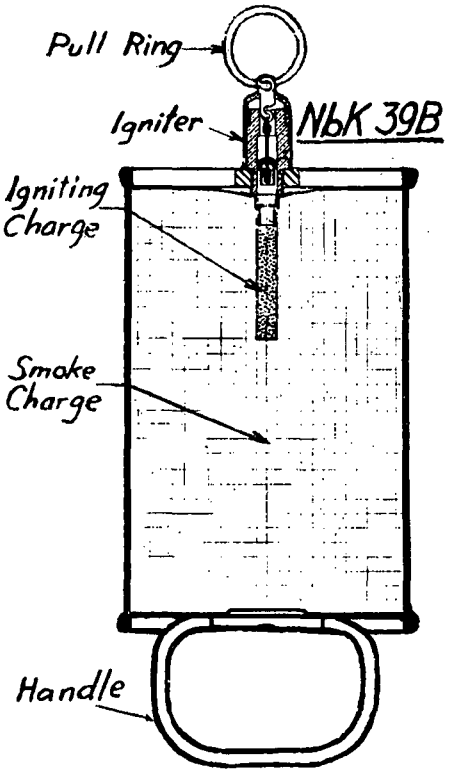
Gemperlé Explosive, patented in England in 1882, was also manufd in Switzerland under the name of *Amidogène* (See also Amidogène in Vol **1**, p A171-R)

The Gemperlé Explosive contained Amm Nitrate 73, bran or starch 8, charcoal 8, sulfur 10 and Mg sulfate 1%. The ingredients were mixed in wet condition and grained and dried. The mixture was suitable as a gun propellant and for blasting operations. It was used for blasting rocks at the Iron Gate on the Danube. Because the powder was hygroscopic, it was necessary to load it into waterproof cartridges
Refs: 1) Cundill, MP **5**, 346 (1892)
2) Daniel (1902), 20 & 340

Generators, Chemical and Smoke Producing. (Apparatus for producing gas or vapor). In Chemical Warfare Service (See CHEMICAL AGENTS in Vol **2**, pp C165-R to C171-R), it refers to a generator for producing poison gas, lacrymatory gas or smoke. For example, the German *Z5 Generator*, originating in France, consisted of a rectangular sheet metal container in which a paper bag was filled with the following liquid mixture: DM (Adamsite) 43.2, Amm Perchlorate 28.5 and urea resin syrup 28.3% (Ref 1). On breaking the container a poisonous gas was emitted. Compn of DM is given in Vol **2**, p C167-R

In a Brit classified Rept (Ref 2) are listed, but not described, generators producing chemical, lacrymatory and smoke gases

Accdg to Bateman (Ref 1), all German smoke generators of WWII examined by him, consisted of a sheet metal container filled with one of the varieties of Berger mixtures. The containers were provided with one or several emission holes. In Berger mixtures hexachloroethane (abbrd as Hexa) was used as a source of chlorine which reacted with powdered Zn or Fe. All smoke compns were ignited by means of an igniter assembly. Some of the smoke generators were called Smoke Candles (Nebelkerze or Rauchkerze). For example, *Nebelkerze 39B* shown here



in Fig was described in Ref 1, pp 10–12, Ref 3, pp Ger 182–83 and earlier by F.G. Haverlack in PATR **1440** (1944). It was a sheet metal cylinder 91mm in diam and 140mm long filled with "Hexa" 59–60 & Zn dust 35–40 with Ba nitrate 1–2% added. Total wt was 4 lb, 2½ oz. For operating the NbK 39B the pull ring of the igniter, connected to a friction wire, was quickly withdrawn thru 0.035g of priming compn contg antimony sulfide 54, K chlorate 33 & MF 13%. The

resulting spark ignited 0.315g of upper mixt: Pb_3O_4 (red lead) 75.4, silicon & fuel with binder 6.6% and a lower layer 1.82g of Pb chromate 50, K perchlorate 23.5, silicon 25.5 & binder 1.0%. After burning for 3 seconds, the smoke charge was ignited and gases generated on burning forced an exit thru the two holes in the steel top. A large vol of grey smoke was emitted for about 3 mins

One of the generators, namely *Parachute Recognition Smoke Generator* was described in TM 9–1985–2 (1953), pp 89–92 and in Ref 3, pp Ger 183–84. Its Fig shown there is not reproduced here because it is too complicated. The device consisted of an Al cylinder, divided into two sections, one housing the canisters with smoke producing compn, while the other the parachute. The smoke compn consisted of a heat-stable blue dye 42 mixed with K chlorate 33 & lactose 25%. As priming compn BkPdr was used. The description of functioning is given on p Ger 184-L of Ref 3

Refs: 1) E.W. Bateman, CIOS Rept (1945), pp 10–12 2) Anon, "Comprehensive List of Government Explosives 1955", British Admiralty Report BR 819 (1B/54) (Conf) 3) Fedoroff et al, PATR **2510** (1958), pp Ger 183–184

Genetron 1132A. Designation of Difluoroethylene or Vinylidene Fluoride, described in Vol **6** under FLUORINE DERIVATIVES, p F126ff

Gentisic Acid and Derivatives

Gentisic Acid or 2,5-Dihydroxybenzoic Acid, $C_6H_3(OH)_2COOH$; mw 154.12, colorless crysts mp 199–200°; sol in w, alc & ether; insol in CS_2, chlf & benz. Can be prepd from hydroquinone, $KHCO_3$ & water at 130°. Used in medicine as Na gentisate

Refs: 1) Beil **10**, 384, (180) & [257] 2) Senhofer & Sarlay, Monatsh **2**, 448 (1881) 3) CondChemDict (1961), 531-R; (1971), 414-L

3-Nitrogentisic Acid, $C_6H_2(NO_2)(OH)_2COOH$; mw 199.12, N 7.03%; crysts, mp 230° (dec). Prepd from $C_6H_3(OAc)_2COOCH_3$ and sp gr 1.52 nitric acid at ambient temp

Refs: 1) Beil, not found 2) A. Klemenc, Monatsh **33**, 1243–54 (1912) & CA **7**, 588 (1913) 3) N.H. Beaugeard & J. Matti, BullSocChFr **1956**, 1612–15 & CA **51**, 7378 (1957)

4-Nitrogentisic Acid, $C_6H_2(NO_2)(OH)_2COOH$; mw 199.12, N 7.03%; yel crysts (from w), mp 242°; mp subl at 180°; sol in alc & hot w; insol in most organics. Prepd from the parent & nitric acid in cold eth

Ref: 1) Beil **10**, (184) 2) F. vonHemmelmayr, Monatsh **34**, 819 (1913) 3) S.C. Bhattacharyya & D.E. Seymour, JCS **1950**, 1139–40 & CA **44**, 10685 (1950)

Dinitrogentisic Acid, $C_6H(NO_2)_2(OH)_2CO_2H$; mw 244.12, N 11.48%, OB to CO_2 –52.5%. There is no mention of this compd in the open literature

Refs: 1) Beil, not found 2) CA, not found

Trinitrogentisic Acid, $C_6(NO_2)_3(OH)_2CO_2H$; mw 289.12, N 14.53%, OB to CO_2 –30.4%. There is no mention of this compd in the open literature

Refs: 1) Beil, not found 2) CA, not found

GEOM. An Ital nonpermissible expl described in Vol **3** of Encycl, p C439-L (Two formulations are listed)

Geophone. An extremely sensitive electromechanical device used as recording instrument in seismic prospecting. See in this Vol, under "Exploration for Natural Gas and Oil by Seismic Method", p E358-L to E359-L

Geophysical Prospecting. See under "Exploration for Natural Gas and Oil by Seismic Methods" in this Vol, p E358-L to E359-L

German Ammunition. Bibliography is listed in PATR **2510** (1958), pp Ger 6 to Ger 7

German Bombs are described, with numerous illustrations in PATR **2510**(1958), pp Ger 14 to Ger 20

German Cannons (Guns). See PATR **2510** (1958), pp Ger 238 to Ger 259, under WEAPONS

German Carbins and Rifles. See PATR **2510** (1958), pp Ger 230 to Ger 237, under WEAPONS

German Commercial (Industrial or Mining) Explosives (Gewerblichesprengstoffe) of WWII. See PATR **2510**(1958), pp Ger 29 to Ger 30, including Table 9; also under GELATIN EXPLOSIVES and under GERMAN DYNAMITS AND THEIR SUBSTITUTES below

German Deep Bonding Process. See Vol 3 of Encycl, pp D37-L to D38-L

GERMAN DYNAMITS AND THEIR SUBSTITUTES

Introduction

Accdg to Stettbacher (Ref 1, p 233), they may be subdivided into:

1) *Guhrdynamit*, which is an obsolete Dynamite with the inactive base Kieselguhr. It usually contained NG 75 & Kieselguhr 25%

2) *Mischdynamite* (Mixture Dynamites), which are nongelatinous (powdery) Dynamites with active base, such ad woodmeal and nitrates, with NG content 12–4%. They correspond to American "Straight Dynamites". The *Ammonsprengstoffe* (Ammonium Nitrate Explosives) belong to this group

3) *Sprenggelatine* corresponds to Amer "Blasting Gelatin"

4) *Gelatine dynamite* corresponds to Amer "Gelatin Dynamites" of which *schwehrgefrierbaren* (difficultly frozen) *Sicherheitsdynamite* (Safety Dynamites) contain as an admixt to NG either NGc, DNClH or aromatic nitrocompds, such as mixtures of DNT with TNT

5) *Wettersichere Dynamite* (gelatinous and nongelatinous) are those safe to use against firedamp and coal dust. They correspond to Amer "permissible" or British "permitted" Dynamites

6) *Pentrinits* are low-freezing mixtures proposed by Dr Stettbacher in 1929. These mixtures consist of NG with 20% or more of PETN (Ref 2, pp 83–4 & 104). Another Dynamite developed by Dr S is *Gelatine-pentrinit.* It consists of Gelatine-Dynamit [such as contg NG with NGc 62.5, Collodion Cotton 2.5, Na nitrate 27 & woodmeal (or rye flour) 8%, with or without prepared chalk (Schleimkreide) up to 0.5%)] to which is added PETN, at least 15%. It is claimed that Gelatine Pentrinit is not affected by "aging" as are other Gelatin-Dynamites (Ref 2, p 85). Ger Dynamites used for rock & ore blasting are known as *Gesteinsdynamite*

German Dynamites used before and during WWII are listed in PATR **2510** (Ref 3) and also in this Encyclopedia. A complete list of A to D compns and description of E to Z compns are given under DYNAMITES AND THEIR SUBSTITUTES:

Alkalsit I, Ammonal, Ammoncahüsit, Ammoncarbonit(Ammonkarbonit), Ammondynamit, Ammongelatine, Ammongelit, Ammonit, Ammon-Nobelit, Ammonpentrinit, Ammonsprenggelatine, Anagon, Argonit, Arit, Astralit, Barbarit, Bautzener-Sicherheitspulver, Bavarit, Berclavit B, Bergmann, Bicarbite (or Bikarbit), Bichel, Bielefeld, Bomlit, Brank (von), Cahusit, Calcinit, Carboazotine, Carbonit, Celtite (or Zeltit), Chemische Fabrik AG Dynamites, Chloratit, Chlorat-Rivalit, Chloratzit, Chrome-Ammonit, Claessen, Cologne (Köln)-Rottweiler Safety Powder, Dahmen, Dahmenit, Detonit, Ditmar's, Donar, Donarit, Dualin, Duxit, Dynamit N, Dynamit 1, Dynamit 2, Dynamit 3, Dynamit 4, Dynamit 5, Dynamite of Trauzl & Abel (also known as Guncotton Dynamite), Dynammon Sprengstoffe, Energit (or Nitroglycerin Powder), Ersatzdynamite, Fördit, Fuchs Powder, Fulmenit and Fulminatin

The following German expls are listed here with prefix **German:**
Gelatin Dynamites (See Ger Gelatine-Astralit to Ger Gelatine-Wetter-Nobelit), Gelignit II, Gesilit, Gestein-Dynamite (See Ger Gesteins-Albit to Ger Gesteins-Westfalit), Glückauf, Glukodine, Gummidynamit, Guncotton or Trauzl Dynamit, Guhrdynamit, Guhrhellhofit, Haloklastit, Hellhoffit or Hellhoffsprenstoff, Heraklin, Hexonit, Hexoplast 75, Kessen-sprengstoffe, Kinetit, Kohlen-Dynamites (See Ger Kohlen-Albit to Ger Kohlen-Westfalit V), Kolax, Kolfit, Kolf's Blasting Powder), Koronit V, Kubin Powder, Lansdorf, Leonit

German Electric Fuzes. See PATR **2510** (1958), pp Ger 40 & Ger 41 with two illustrations

German Fillers (Bursting Charges) for Projectiles. See German Military Explosives of WWII

German Flares. See PATR **2510**(1958), pp Ger 49 to Ger 51 with illustrations

German Fuzes. A comprehensive description with numerous illustrations is given in PATR **2510**(1958), pp Ger 54 to Ger 64

German Gelatine. See under GELATIN in this Vol

German Gelatine Dynamits are listed in this Volume under their individual names, such as Gelatine Astralit (Ger), Gelatine Carbonit (Ger) and under Gelatine-Dynamits (Ger). See also PATR **2510**(1958), pp Ger 66-L to Ger 68-L

German Gerlich Projectile. See German Tapered Bore Gun Projectile

German "G" Propellant ("G" Pulver), developed before WWII by General Uto Gallwitz and collaborators is described in PATR **2510** (1958), pp Ger 70-R to Ger 71-R and in Vol **5** of Encycl, p D1536-R

German Granaten (Grenades, Projectiles, or Shells) are described with numerous illustrations in PATR **2510**(1958), pp Ger 71-R to Ger 81-L

German Gudol Propellant (Gudolpulver) is described in PATR **2510**(1958), p G81-L & R and in Vol **5**, pp D1537-R to D1538-R

German Guidance Systems and Guidance Missiles are described with numerous illustrations in PATR **2510**(1958), pp Ger 81-R to Ger 85-L and in this Vol

German Guns and Cannons. See PATR **2510** (1958), pp Ger 238 to Ger 259 & Ger 263, under Weapons

German Gun Propellants of WWII. The brief info given below is from PATR **2510** (Ref), pp Ger 140 to Ger 147, which contains the description of results of analysis of captured German proplnts, conducted at Picatinny Arsenal, Dover, New Jersey (mostly by P.R. Hosken, Jr & H. Jadowitz of the General Laboratory) and also from documentary materials gathered by various American and British missions sent to Germany directly after termination of the War. The work conducted at PicArsn was summarized by A.B. Schilling and others in PATR's listed on p Ger 140-R of Ref. The reports of Amer & British missions are listed on the same page

In Tables included in Ref are listed 26 Single-Base (NC) Propellants, 23 Double-Base (NC-NG) Propellants, 41 Double-Base (NC-DEGDN) Propellants, 11 Triple-Base

(NC-DEGDN-NGu) Propellants and Artillery Propellants described by H.H.M. Pike in CIOS Rept **31-68**(1946). In Table 46 on p Ger 146 are listed ballistic properties and uses of several propellants. Abbreviations used in Tables: DEGDN means Diethyleneglycol Dinitrate and NGu is Nitroguanidine, called Picrite in GtBritain. Many of the German proplnts of WWII were original and not known in other countries. The most important of these proplnts were "cool" propellants developed, beginning in 1934, under the direction of General Uto Gallwitz. These NC-DEGDN proplnts known as "G" Pulvern (or "K" Pulvern) are briefly described in Vol **3** of Encycl, pp C511-R to C512-L and in Vol **5**, p D1536-R. For uses in hot climates (such as in Africa), DEGDN was replaced by TEGDN (Triethyleneglycol Dinitrate). Some of these proplnts, known as DiglP and TriglP are listed in PATR **2510**, p Ger 147-L

The "cool" Triple-Base (NC-DEGDN-NG) Propellants, known as GuP (Gudolpulvern) are described in Vol **5**, pp D1537-R & D1538-L, and also in PATR **2510**, p 147-L

Ref: B.T. Fedoroff et al, PATR **2510**(1958), pp Ger 140 to Ger 147

German Handgrenades. See PATR **2510** (1958), p Ger 86 (Illustrated)

German HDP Supergun [Hochdruckpumpe, V-3 (Vergeltungswaffe 3), High Pressure Pump, "Busy Lizzie" or "Multipede"]. See PATR **2510**(1958), p 90 (Illustrated)

German Hollow Charges (Hohlladungen) or Shaped Charges. See PATR **2510**(1958), pp Ger 91-L to Ger 93-L (Illustrated)

German Igniters. See PATR **2510**(1958), pp Ger 93-R to Ger 99-L (Illustrated)

Germanium, Gadolinium and Gold Salts were proposed as ingredients in smoke producing compns

Ref: J. DeMent, USP 2995526(1961)

German Land Mines (Landminen). See PATR **2510**(1958), pp Ger 104-L to Ger 107-L (Illustrated)

German Long Range Guns. See Big Bertha, Paris Gun and Other German Big Guns of WWI and WWII in Vol **2** of Encycl, pp B113-R to B114-L. Also in PATR **2510**(1958), pp Ger 258 & Ger 259, under Weapons

German Man Salt (MAN-Salz). See PATR **2510**(1958), pp Ger 108-L to Ger 109-R

German Markers. See PATR **2510**(1958), pp Ger 110 & Ger 111 (Illustrated)

German Message Tube (Meldebüchse). See PATR **2510**(1958), p Ger 112

German Metriol Trinitrate. See PATR **2510** (1958), p Ger 113-L

GERMAN MILITARY EXPLOSIVES OF WWII. The expls used as bursting charges (fillers) for bombs, shells and grenades were known under the name *Füllung* or *Füllpulver* (abbrd as Fp), followed by some number, signifying mostly the year of adoption for Service. For example: Fp 02 signified TNT, which was adopted in 1902. Some of the fillers were not abbrd as Fp, but as Grf (Granatefüllung), as, for example Grf 88 signified Picric Acid. It was adopted in 1888 for filling shells

In PATR **2510** (Ref 4) are listed 56 fillers, compiled from Refs 1 to 3

Refs: 1) Anon, "Recognition Handbook for German Ammunition", Supreme Headquarters

Allied Expeditionary Force (1945), pp 286–88 2) Anon, "Allied and Enemy Explosives", Aberdeen Proving Ground, Maryland (1946), pp 75, 79, 82, 86, 88, 97, 112, 113, 118, 120, 124, 129, 133, 134, 137, 139, 141, 142 & 147 3) Anon, US Dept of the Army Tech Manual, **TM 9–1985–3** (1953), pp 536–37 4) B.T. Fedoroff et al, PATR **2510**(1958), pp Ger 146-R to Ger 148-R

German Military Explosives of WWII Used as Fillers in Anticoncrete and Armor-Piercing Shells. In order to make the explosives such as TNT safe for use in armor-piercing and anticoncrete shells, sections of TNT close to the nose were made less sensitive to shock by incorporating some wax & K chloride

A good example of this type of filling was the one in 210mm Anticoncrete Shell (21cm GrBe). Its filler consisted of ten pressed pellets placed in cardboard container and held in position by a cement lining. The forward three sections 6, 7 & 8 were intended to provide protective layers, ptactically insensitive to shock, whereas the layers close to the base were nearly or just as sensitive as straight TNT. The following list gives the compositions and weights of charges shown in Fig G14

No 1 4 lb, 2 oz of TNT/Wax – 94/6
No 1a 8 oz of Straight TNT
No 2 4 lb, ¾ oz of TNT/Wax – 90/10
No 2a 1 lb, 5½ oz of Straight TNT
No 3 5 lb, 5½ oz of TNT/Wax – 90/10
No 4 5 lb, 4½ oz of TNT/Wax – 91/9
No 5 4 lb, 2 oz of TNT/Wax – 91/9
No 6 6 oz of TNT/Wax/KCl–60.5/5.4/34.1
No 7 5 oz of TNT/Wax/KCl–44.1/5.6/50.3
No 8 6 oz of KCl
Total weight of filler was 25 lb, 8¾ oz
Refs: 1) E. Englesburg, The Ordnance Sergeant, May 1944, p 320 2) PATR **2510** (1958), p Ger 48-R

German Military Powders. A name sometimes applied to Ger Ammonals

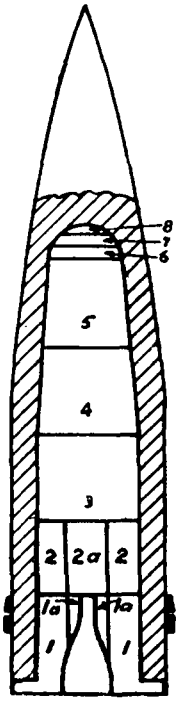


FIG G14

German Mine Explosive of WWI. Due to the shortage of NG, some mine expls contd no such substance, as for example: DNB 32, DNN 12 & K perchlorate 56%
Ref: Davis (1943), 158

Germen Minenhund (Mine Dog), called by the Allies "Doodlebug" or "Goliath". See PATR **2510**(1958, p Ger 113-L

German Muzzle Charging Device. See PATR **2510**(1958), p Ger 114

German Myrol and Its Explosives. See PATR **2510**(1958), pp Ger 114-R to Ger 116-L

German Natter Ba349A & Ba349B Guided Missiles. See PATR **2510**(1958), p Ger 116-R (Illustrated)

German Naval Explosives and Weapons of WWI and WWII were described by A. Stettbacher in Protar (Switzerland) **9**, 33–45(1943). The paper was entitled: "Ueber die Wirkung von Torpedos, Minen, und Tiefbomben unter Berücksichtigung der deutschen Marinesprengstoffe vom letzten und heutigen Weltkrieg" (On the Work of Torpedoes, Mines, and Depth Charges in Regard to German Naval Explosives from the Last and Present World War)

During WWI Ger *Whitehead torpedoes* were loaded with **Hexyl**, which consisted of TNT 60 & HNDPhA (Hexanitrodiphenylamine) 40%. This was replaced during WWII with TNT 55.7, HNDPhA 27.9 & Al (grit 40–70 sieve) 16.4%. Another Ger compn of WWII contd (accdg to analysis of Dr Stettbacher) TNT 61.8, HNDPhA & Al 15.2%. The torpedoes were cigaret-like steel vessels 45–60cm diam and 5–10 meters long. They consisted of 3 compartments, of which the head (Kopf), also called warhead, contained the expl

Straight PETN was found to be too sensitive for loading torpedoes

Ger *sea mines* (Seeminen) were of the following types: *contact mine* (Kontaktmine or Stoss- und Streumine), *magnetic* and *acoustic* mines. PETN, straight or in mixt with Al, was used as expl chge for mines

Ger *depth bombs* (Tiefbomben or Wasserbomben) were used against submarines. A typical bomb consisted of a metallic cylinder ca 50cm diam and 80cm long. It contd ca 350kg of expl, such as TNT, PA or aluminized TNT–HNDPhA

German Nipolit Propellants. See PATR **2510**(1958), p Ger 117

German Nonpermissible Explosives. See Vol **3** of Encycl, p C438-R

German Observing Bullet. See PATR **2510**(1958), pp Ger 122-R to Ger 123-L (Illustrated)

German One-Man Torpedo. See German Torpedo, One Man

German Panzer (Armor or Armed Vehicle; Tank). See PATR **2510**(1958), pp Ger 123-L to Ger 126-R

German Panzerfaust and Panzergranate. See PATR **2510**(1958), pp Ger 126-R to Ger 127-L (Illustrated)

German Panzerschreck and Panzerwurfmine. See PATR **2510**(1958), p Ger 127 (Illustrated)

German Permissible Explosives. See Vol **3** of Encycl, p C451-L

German Photoflash Bomb and Photoflash Composition. See PATR **2510**(1958), pp Ger 130-R & Ger 131-L (Illustrated)

German Pistols of WWII. See PATR **2510** (1958), pp Ger 227 to Ger 229 (Illustrated)

German Pistol Grenades. See PATR **2510** (1958), pp Ger 132-R to Ger 134-L (Illustrated)

German Primary Compositions and Primers. See PATR **2510**(1958), pp Ger 137-L to Ger 138-R (Illustrated)

German Propagandarakete (Leaflet Rocket). See pp Ger 138-R & Ger 139 (Illustrated)

German Propellants. See Gun Propellants on pp Ger 139-L to Ger 149 and for Rocket Propellants, pp Ger 166-R to Ger 168-L

German Propellent Igniters. See PATR **2510**(1958), p Ger 151-L

German Proximity Fuze. See PATR **2510** (1958), p Ger 151-R (Illustrated)

German Püpchen or Wheeled Bazooka. See PATR **2510**(1958), p Ger 152-R (Illustrated)

German Pyrotechnic Antipathfinder Devices. See PATR **2510**(1958), pp Ger 152-R & Ger 153-R

German Pyrotechnics. See PATR **2510**(1958) pp Ger 153 to Ger 155-R

German R-4M Rocket. See PATR **2510** (1958), p Ger 155 (Illustrated)

German Recoilless Gun (Kanone ohne Rückauf). See PATR **2510**(1958), p Ger 156-R

German Rheinbote (Unguided Missile). See PATR **2510**(1958), p 157-L (Illustrated)

German Rheintochter (Guided Missile). See PATR **2510**(1958), p Ger 157-R

German Rifle (Gewehr). See PATR **2510** (1958), pp Ger 229 to Ger 237, under Weapons

German Rifle Discharger. See Schiessbecher in PATR **2510**(1958), p Ger 172-L (Illustrated)

German Rifled Projectile. See PATR **2510**(1958), p Ger 157-R

German Rifle Grenades. See PATR **2510** (1958), pp Ger 158-L to Ger 160-L

German Röchling Projectile. See PATR **2510**(1958), p Ger 160-R (Illustrated)

German Rocket (Rakete). See PATR **2510** (1958), pp Ger 160-R to Ger 163-R (Illustrated)

German Rocket-Assisted Shell. See PATR **2510**(1958), pp Ger 163-R to Ger 164-L (Illustrated)

German Rocket Launchers. See PATR **2510**(1958), pp Ger 164-L to Ger 165 (Illustrated)

German Rocket Propellants of WWII. Accdg to Urbański (Ref 1), the Germans used solid double-base (NC-NG) proplnts in their smaller rockets. The larger types, such as the V-2 (described in Ref 3, p Ger 213-R), used liquid propellants consisting of a fuel (such as hydrazine, alcohol, fuel oil, etc) and an oxygen carrier (such as hydrogen peroxide, nitric acid, Tetranitromethane, etc). Mixtures of easily oxidizable organic liquids with H_2O_2 of 80–85% strength were the most widely used. Hydrogen peroxide was also used as the driving force without any fuel, because the heat liberated accdg to the reaction of decompn: $H_2O_2 \rightarrow H_2O + \frac{1}{2}O_2 +$ 23450kcal, was sufficiently great. The liberated water vapor and oxygen served as driving force

Liquid proplnts were also used for driving V-1, described in Ref 3, p Ger 213-L with Fig on p Ger 214

Accdg to Tschinkel (Ref 2), some V-2 rockets were driven by a mixt of liquid oxygen with alcohol contg some w, which was added in order to keep the flame temp as low as possible to avoid damage to the

combstn chamber. Incorporation of 25% w in alc lowered the flame temp 7%, while exhaust velocity of gases was lowered only 3.5%. In 1944 prepns were made to replace liq oxygen with absol nitric acid

Table 53 of Ref 3, p Ger 167 lists six formulations of solid rocket proplnts, while Table 56 lists five formulations of WASAG-AG, as reported in PB and OSRD Repts listed on p Ger 168-L

Refs: 1) T. Urbański, Przemysl Chemiczny **27**(4), 187(1948) 2) J.G. Tschinkel, C&EN **32**, 2584(1954) 3) PATR **2510** (1958), p Ger 167

German Rodded Bomb or Stick Grenade. See PATR **2510**(1958), p Ger 168 (Illustrated)

German R-Salt (R-Salz) and Its Explosives. See PATR **2510**(1958), p Ger 170

German Sabot Projectile. See PATR **2510** (1958), pp Ger 170 & Ger 171 (Illustrated)

German Sarin. See under German Trilons and GB in Vol **2** of Encycl, p C167-R

German Sänger-Bredt Supersonic Missile See PATR **2510**(1958), p Ger 171-R

German Schnorkel or Snorkel. See PATR **2510**(1958), pp Ger 172-R & Ger 173-L

German Sea Dog (Seehund). See PATR **2510** (1958), p Ger 174-L

German Self-Destroying Fuze, Tracer Bullet and Tracer Shell. See PATR **2510**(1958), p Ger 176-L (Illustrated)

German Sebastopol Gun, cal 800mm, nicknamed *Dora* and *Gustav Geschütz.* See PATR **2510**(1958), p Ger 176 and p Ger 259, under Weapons

German Shrapnel Projectiles. See PATR **2510**(1958), pp Ger 176-R to Ger 177-R

German Signal Device. See PATR **2510** (1958), pp Ger 177-R to Ger 179-L

German-Sinoxyd Priming Mixture. See PATR **2510**(1958), p Ger 179-R

German Small Arms Ammunition. See PATR **2510**(1958), pp Ger 180-L to Ger 181-L

German Smoke and Chemical Rocket. See PATR **2510**(1958), p Ger 181-R

German Smoke Bomb. See PATR **2510**(1958), pp Ger 182-L to Ger 183-R (Illustrated)

German Smoke Generator (Rauchentwickler). See PATR **2510**(1958), pp Ger 183-R to Ger 184-L

German Smoke Hand Grenade (Blendkörper). See PATR **2510**(1958), pp Ger 184-L to Ger 185-L (Illustrated)

German Smoke Projectile (Nebelgeschoss). See PATR **2510**(1958), pp Ger 185-L to Ger 186-L (Illustrated)

German Soman. See under German Trilons in this Vol and GD in Vol 2 of Encycl, p C168-L

German Sound Gun. See PATR **2510**(1958), pp Ger 186-R to Ger 187-R (Illustrated)

German Spigot Mortar Projectile. See PATR **2510**(1958), pp Ger 187-R to Ger 188-L (Illustrated)

German Spike Bomb. See Stachelbombe, abbrd Stabo, in PATR **2510**(1958), p Ger 190 (Illustrated)

German Spotting Projectile. See PATR **2510** (1958), pp Ger 188-L to Ger 189-L (Illustrated)

German Sprengbrandbombe. See PATR **2510** (1958), p Ger 189-L (Illustrated)

German 132° Stability Test. This test has been used in Germany for testing NC and powders. The test was formerly carried out at 135°, but the temperature was lowered to 132° and the observation was often confined to the visible brown fumes. Sometimes the test was run using a strip of litmus paper. The test is essentially the same as the German 135° Stability Test (qv)
Refs: 1) Reilly (1938), p 82 2) Kast-Metz (1944), p 233

German 135° Stability Test. This test originated in Germany about 70 years ago and was described by Sy (Ref 1, p 556). It was adopted by the US Ordnance Dept (Ref 2), but the temperatures of the test were 120° for double-base propellants and 134.5° for single-base propellants & for NC's (Ref 3). Litmus paper was originally used in this test (Refs 1 & 2) but later this was changed to methyl violet paper and the test was called the "Methyl Violet Test"
Apparatus:
A cylindrical constant temperature bath containing glycerin-water mixture of density 1.21 for 120±0.5° and d 1.24 for 134.5±0.5°. The top of the bath is perforated and equipped with several metallic tubes about 285mm long and about 20mm ID, which extend into the liquid of the bath and serve to sheath the Pyrex test tubes. A calibrated thermometer is inserted in the glycerin-water mixture

Heavy-walled Pyrex tubes 15mm ID, 18mm OD and 290mm long, are provided with ordinary cork stoppers; the side of each is notched to a depth of about 4mm

Normal methyl violet test paper is prepd by soaking filter paper in a normal solution of rosaniline acetate-crystal violet and drying
Note: Standard methyl violet paper could be purchased from US Naval Powder Factory, Indian Head Maryland, changed to Naval Propellant Plant (NPP)
Procedure for NC and for Propellants
a) Weigh out two (or three) 2.5g portions of the sample (previously dried either for 4–5 hrs at 40±2° or for 1–1½ hrs at 50±2° or overnight at RT with further drying for ½ hr at 40±2°) and press them into the lower part of the Pyrex test tubes
b) Place a piece of standard normal methyl violet paper, 70mm long and 20mm wide, vertically in each tube, its lower edge being 25mm above the test material; stopper the tubes
c) Place each Pyrex tube into a metallic tube extending inside the bath maintained at 134.5±0.5° and observe the time
Note: No more than 6–7mm of either of the Pyrex tubes should project above the top of the bath
d) After 20 minutes of heating, examine each tube at five minute intervals by withdrawing one-half its length. Replace the tubes as quickly as possible to avoid cooling more than necessary
e) Record as a *salmon pink* (SP) value, the time in minutes required for the test paper completely change from violet to salmon pink.

When testing smokeless propellants, heating may be continued in order to determine the time at which visible "red fumes" (RF) are evolved. The test is usually also extended to a total of 5 hours heating in order to determine if the proplnt explodes

in less than 5 hours (300 minutes)

On testing proplnts, each sample should contain as many whole grains as possible but, if the grains are too large, they should be sectioned longitudinally

Requirements of this test vary with the proplnts and are given in specifications. Generally, minimum "salmon pink" time is 30 minutes for NC, 45 mins for single-base proplnts and 40 mins for double-base proplnts

Note: More detailed description as conducted at PicArsn is in Ref 3

Refs: 1) A.P. Sy, JACS **25**, 553 & 556 (1903) 2) Marshall **2**(1917), 662 3) A.J. Clear, PATR **3278**(1965), pp 23 to 25; (1970), 24–25

German Star Shell. See PATR **2510**(1958), pp Ger 190-R & Ger 191-L (Illustrated)

German Steel and Iron Ammunition Items. See PATR **2510**(1958), p Ger 191 (Illustrated)

German Substitute Explosives of WWII. See Ersatzsprengstoffe in Vol **5** of Encycl, pp E121 & E122 (Table E15)

German Supergun. See German HDP Supergun (Hochdruckpumpe or V-3)

German Tabun. See under German Trilons and GA in Vol 2 of Encycl, p C167-R

German Taifun Rocket, See PATR **2510** (1958), p Ger 193-L

German Tapered Bore Gun (Würgebohrung Geschütz). See PATR **2510**(1958), p Ger 193

German Tapered Bore Projectile or Gerlich Projectile. See PATR **2510**(1958), pp Ger 193-R to Ger 195-R (Illustrated)

German Tanks. See German Panzer in PATR **2510**(1958), p Ger 123 to Ger 127-L

German Tarbun. See under Trilons

German Tellermine (Dish-like Land Mine). See PATR **2510**(1958), p Ger 195

German Tetan (TeNMe) or X-Stoff and Its Explosives. See PATR **2510**(1958), pp Ger 195 & Ger 196

German "Thor" and "Karl" Mortars. See PATR **2510**(1958), p Ger 198-L

German Tiger Tanks. See PATR **2510**(1958), p Ger 126-L, under Panzer

German Torpedo, One Man. See U-Boat, One Man in PATR **2510**(1958), p Ger 211-L (Illustrated)

German Torpedoes, Mines and Depth Bombs of WWI and WWII were described by A. Stettbacher in Protar (Switzerland) **9**, 33–45 (1943)

German Totalits. See PATR **2510**(1958), p Ger 199-R

German Tracer Compositions and Tracers. See PATR **2510**(1958), p Ger 199-R to Ger 202-L

German Tracer Projectiles. See PATR **2510**(1958),pp Ger 202-L to Ger 203-L (Illustrated)

German Trilons. Extremely toxic Chemical Warfare Agents developed before WWII but never employed. They included *Sarin, Soman, Tabun* and probably others. See PATR **2510**(1958), p Ger 204-L and GA and GB in Vol 2 of Encycl, p C167-R

German T-Stuff (T-Stoff). Concentrated Hydrogen Peroxide. See PATR **2510**(1958), p Ger 210

German U-Boat-21 (Unterseeboot-21). See PATR **2510**(1958), p Ger 211-R

German U-Boat, One Man. See German Torpedo, One Man

German U-Boat Walter. See PATR **2510** (1958), p Ger 211-R

German V-1 (Vergeltungswaffe Eins)(Revenge Weapon One). German designation FZG-76; British name *Buzz Bomb.* See PATR **2510**(1958), p Ger 213 (Illustrated on p Ger 214)

German V-2 (Vergeltungswaffe Zwei) (Revenge Weapon Two). See PATR **2510**(1958), p Ger 213 (Illustrated).

German Vocabulary of Ordnance, etc. See PATR **2510**(1958), p Ger 265 to Ger 345

German Warplants, Arsenals, Research Centers and Proving Grounds. See PATR **2510**(1958), pp Ger 217 to Ger 225

German Weapons of WWI and WWII. See PATR **2510**(1958), pp Ger 227 to Ger 259 & Ger 263. Also R. Lusar, "Die Deutschen Waffen und Geheimwaffen des 2 Weltkrieges und Ihre Weiterentwicklung", Lehmann Verlag, München (1958)

German (West) Weapons. These include: 1) Redesigned MG-42 machine gun firing 7.62mm NATO rounds; 2) Fully automatic Spanish Cetme rifle, also 7.62mm, firing at a cyclic rate of 650 rounds/min and 3) 20mm automatic cannon by Hispano Suiza, firing 800 rounds/min at a muzzle velocity of 1050m/sec

Ref: J. Weller, Ordn **45**, pp 351–354(Nov-Dec, 1960)

German Wettersprengstoffe (Permissible Explosive). Definition is given in PATR **2510**(1958), p Ger 226 and list with compns in Table 64 on pp Ger 260 & Ger 261. Properties of some Wettersprengstoffe are in Table 65 on p Ger 261. Notes and Refs are on p Ger 262

German Wind Gun. See PATR **2510**(1958), p Ger 262 (Illustrated)

German Wind Tunnel. See PATR **2510**(1958), p Ger 262-R

German X-4 Guided Missile. See PATR **2510** (1958), p Ger 264-L

German X-Stuff. See German Tetan (TeNMe)

German Z-Salt (Z-Salz). See PATR **2510**(1958), p Ger 264-R

German Z-Stuff C and N (Z-Stoff C & N). See PATR **2510**(1958), p Ger 264-R

Gerresdorfer and Bals Powders, patented in 1892 in France, used Na chlorate as a base, but in order to diminish its hygroscopicity it was coated with an alcoholic soln of a resin mixed with Mn dioxide and with one or several other substances, such as K chlorate, K chromate, sulfur, gum & charcoal
Refs: 1) Daniel (1902), 341 2) Giua, Trattato **6**(1), (1959), p 395

Geserick of Rotterdam, patented in 1895 a permissible expl consisting of AN 88.65, DNBz 7.50, MNNaphthalene 0.50, chloronaphthalene 2.35 & NC 1.00%
Ref: Daniel (1902), 341

Gesilits. German permissible WWI expls: **No 1** – NG 30.75, DNT 5.25, Na nitrate 18.00, dextrin 39.00 & NaCl 7.00%; **No 2** – NG 30.75, DNT 5.25, AN 22.00, dextrin 21.00 & NaCl 21.00%
Refs: 1) E. Colver, "High Explosives", Van-Nostrand, NY (1918), p 167 2) F.M. Turner, Edit, "Condensed Chemical Dictionary", Reinhold, NY (1942), p 289 (Not described in later editions) 3) PATR **2510**(1958), p Ger 68-R 4) Giua, Trattato **6**(1), 1959, p 345

Gesteinssprengstoffe. German for Rock Blasting Explosives, which are listed below. They are suitable for blasting rocks, ores, potash concrete, etc, but unsuitable for use in coal mines contg in atmosphere firedamp and coal dust

Following is a partial list of Gesteinssprengstoffe:
Gesteins-Albit. See Vol **1**, p A120-L
Gesteins-Dorfit. See Dorfit in Vol **5**, p D1535-L
Gesteins-Koronit T1: Na chlorate 72.0, vegetable meal 1.0–2.0, TNT+DNR 20.0, paraffin 3.0–4.0 & NG 3.0–4.0% (Ref 1)
Gesteins-Koronit T2: Na chlorate 75.0, vegetable meal 1.0–2.0, TNT+DNT 20.0 & paraffin 3.0–4.0% (Ref 1)
Geeteins-Permonit: K perchlorate 30.0, AN 40.0, Na nitrate 7.0, TNT 15.0, flour 4.0, woodmeal 3.0 & jelly 1.0% (Ref 2)
Gesteins-Persalit 1: K perchlorate 35.0, AN 43.0, DNT 8.0, DNN 8.0, NG 2.0 & woodmeal 4.0% (Ref 2)
Gesteins-Persalit 2: K perchlorate 34.0, AN 48.0, DNT 10.0, carbon (powder) 2.0 & woodmeal 6.0% (Ref 2)
Gesteins-Westfalit: AN 84.5, DNT 12.0 & Al 3.5% (Ref 2)
Refs: 1) Naoúm, NG (1928), 428 2) PATR **2510**(1958), p Ger 69

Gheksamony (Hexamons). Russ permissible expls based on 8–10% normally or finely dispersed RDX (Gheksogen in Russ) and powd AN (Ammiachnaya Selitra, in Russ). Gheksamony Nos 1, 3, 5 & 6 are listed in Refs, but their exact compns are not given in CA's
Refs: 1) V.G. Khotin et al, VzryvnoyeDelo **1970**(68/25), 243–50 & CA **73**, 94064(1970) 2) V.F. Starkozhev & V.P. Vasil'eva, Ibid, pp 250–55 & CA **73**, 95075(1970) 3) Fedoroff & Sheffield, Encycl, Vol **5**(1972), p D1744-L

Gheksoghen (Hexogen). Russ for RDX

Ghinijonet used to manuf at Ougrée, Belgium a permissible mining expl, *Densite,* described in Vol **3** of Encycl, p 64-R. He also manufd *Tritorite,* which consisted of AN 70, DNBz 18, K nitrate 11 & charcoal 1%
Ref: Daniel (1902), 341 & 775

Giant Coal Mine Powder and Giant Low Flame Powder (Amer). Accdg to Naoúm (Ref) they were permissible Dynamites which contained hydrated salts and, for this reason, were called *Hydrated Explosives.* No compn is given in Naoúm, but it seems that they were similar to Belg & French Dynamite-grisoutites as, for example, NG 42, $MgSO_4.7H_2O$ 46 & woodmeal (or guhr) 12%
Ref: Naoúm, NG (1928), 399

Giant Powder. Under the title *Géante (Poudre),* Daniel (Ref 1) called this an older Amer name for *Dynamite,* manufd in Calif under the name *Dynamite-Lignine,* which contd Na nitrate. Accdg to Naoúm (Ref 4), the name *Giant Pow-*

der No 1 was applied to Dynamite No 1, which was *Guhr Dynamite* consisting of NG 75 & Kieselguhr 25%

Accdg to Daniel (Ref 1), *Giant Powder No 2* contd NG 40, Na and/or K nitrate 40, rosin 6, sulfur 6 & kieselguhr 8%. The ssme compn for Giant Powder is given by Ramsey & Weston (Ref 3). Gody (Ref 2), under the name *Poudre géant* gives: NG 36, saltpeter 48, sulfur 8 & rosin or charcoal 8%

Refs: 1) Daniel (1902), 324 2) Gody (1907), p 363 3) Ramsey & Weston (1917), p 21 4) Naoúm, NG (1928), p 264

Giedyuim Powder, patented in 1868, consisted of K chlorate, sulfur & silicon

Ref: Giua, Trattato **6**(1)(1959), p 392

GI Explosive. Abbr for Govemment Issue Explosive

Gilding Metal. Accdg to Gardner (Ref 2) it is a reddish-yellow metal consisting of Cu 70, Sn 12.5 & brass 17.5%

Gilding metal is used for some military purposes. The requirements of US Armed Forces for gilding metal, called "95/5 Brass", are covered by Specification JAN-G-439 (Aug 1946): Cu 94–95 & Zn 6–5% (minus allowable impurities 0.13%, which include Pb max 0.03 & Fe max 0.05%). This type of gilding metal is used as casings for primers and detonators for artillery ammunition and bombs

Gilding metal "90/10 Brass" consists of Cu 89 to 91 & Zn 11 to 9% (minus allowable impurities)

There is also gilding metal "98/2 Brass"

Refs: 1) B. Grotta, ChemMetalEngrg **26**, 1132 (1922) (Gilding metal used for detonator casings contd: Cu 90 & Zn 10%) 2) W. Gardner, "Chemical Synonyms and Trade Names", VanNostrand, NY (1948), p 236

Giles Flask and Its Applications. Giles flask at x and (x+x/10) of its volume, eg, at 500ml and 550ml. Used in prepg normal solns (Ref 1, p 376-R)

We are including here the description of use of Giles flask in analysis of mixed acids by aliquot method, as was done by Clift & Fedoroff (Ref 2, Chap I, Part I, pp 1 to 4) at Triton Chemical Corp, Glen Wilton, Va and at Keystone Ordnance Works, Meadville, Pa

THE ANALYSIS OF COMMERCIAL MIXED ACIDS

(Mixtures of Sulfuric and Nitric Acids)

I. Preparation of Aliquot Solution

A. Apparatus:

Separatory funnel with 60° angle and 1000ml capacity

Giles flask, 1000ml capacity

Support, with ring

Weighing bottle of 15–25ml capacity

Rod with hook to remove weighing bottle stopper

B. Procedure:

1) Arrange the apparatus accdg to Fig G15 with the funnel half filled with water (distilled)

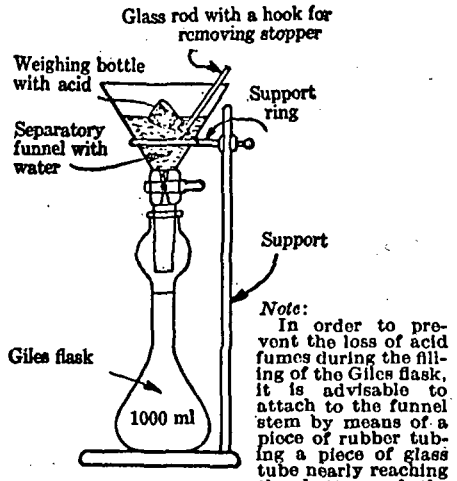


FIG G15

2) Shake the sample of acid to thoroughly mix it and transfer 10ml by means of a pipette into a previously tared weighing bottle

3) Weigh the bottle with the contents. Holding the bottle by two fingers of the left hand plunge the upper part of it, including the cover, into water, taking care not to wet the fingers. To prevent the loss of fumes, remove the stopper of the bottle under the surface of the water by

to displace the acid in the bottle very slowly (See Fig G15)

4) Empty the funnel into the Giles flask

5) Wash 2–3 times the funnel, the stopper, the rod and the outside of the weighing bottle, draining the washings into the flask

6) Wash the inside of the weighing bottle 2–3 times, emptying into the funnel and then into the flask

7) Remove the funnel and fill the Giles flask to the 1000ml mark

8) Shake the flask for 2 min to thoroughly mix the solution which will be used for the determination of total sulfuric acid and total acidity

II. Total Sulfuric Acid

9) Take two 100ml aliquot portions and transfer them into two shallow evaporating dishes (of about 500ml capacity) (For transferring the soln either a pipette or a volumetric flask with a short neck, graduated to deliver 100ml is used)

10) Place the dishes on a steam or water bath to evaporate the water and nitric acid

11) The heating is continued until the disappearance of nitric fumes, which requires about two hours

12) After the evaporation, in order to decompose the nitrosyl-sulfuric acid always present in commercial mixed acids, wash down the sides of the evaporating dish with about 1 ml of distilled water from a wash bottle or pipette

13) Continue the evaporation until the disappearance of fumes (about 15–20 mins)

14) If the nitrosyl-sulfuric acid content is high (more than 1%) as determined by permanganate (see further) add another 1 ml of water and evaporate again

15) The dish, containing the sulfuric acid is removed from the bath and the sides washed down with about 50ml water. The second dish is used as a check

16) A stirring rod is placed in the dish with 3 drops of Methyl Red Indicator

17) Titrate with standard NaOH soln, approx N/3, until the color changes to greenish-yellow representing a pH of about 6.3

18) Use as a color comparison a blank that has been run on an equivalent amount of the distilled water used in the determination, using Methyl Red as indicator

Calculations for Total Sulfuric Acid (TS):

If R is the burette reading, W is the weight of acid in 100ml of soln, NF is the normal factor of the NaOH soln, 49.043 is the equivalent weight of H_2SO_4(½ of mol wt), then the percentage of sulfuric acid as H_2SO_4 is determined by the following formula:

$$TS = \frac{(NF) \times 0.049043 \times R \times 100}{W}$$

III. Total Acidity (TA):

19) Take two 100ml portions of the soln in the Giles flask (see 9) and transfer them into two 400ml conical flasks or beakers

20) Titrate the soln in the same manner as for total sulfuric using Methyl Red indicator

Note: Methyl Red is partly decolorized by nitrous acid, hence it is advisable to add the indicator towards the end of the titration

Calculations for Total Acidity in Terms of H_2SO_4:

$$TA = \frac{(NF) \times 0.049043 \times R \times 100}{W}$$

IV. Determination of Nitrosyl-sulfuric Acid (Nitroso):

21) While the evaporation of the aliquot portions proceeds, take 10ml of the acid sample, using the same pipette that was used for preparing the aliquot soln

22) Transfer the acid to a 400ml conical flask or beaker containing about 100ml of distilled water. (To prevent the heating of the water and the escape of acid fumes the tip of the pipette should be kept in circular motion and held as close as possible to the surface of the water)

23) Run into it from a burette a 0.1–0.2N soln of K permanganate soln until the appearance of the first permanent pink color

Notes:

a) The following reaction takes place during this titration:

$$2KMnO_4 + 5HNOSO_4 + 2H_2O = K_2SO_4 + 2MnSO_4 + 5HNO_3 + 2H_2SO_4$$

b) If a few drops of sulfuric acid are added to the stock permanganate soln, practically no change occurs in the titer, during several months

Calculations for Nitroso ($HNOSO_4$):

If NF is the normal factor of the permanganate soln, and 63.54 is the equivalent weight of $HNOSO_4$, then

$$\%HNOSO_4 = \frac{0.06354 \times (NF) \times R \times 100}{W}$$

Note: The weight of the mixed acid, W, is the same weight as was obtd for the 10ml sample weighed in item 3

Example of Calculation for a Mixed Acid

The total acidity as H_2SO_4, the total sulfuric as H_2SO_4, and the nitrosyl-sulfuric acid having been determined by titration, the HNO_3 and H_2O are calculated by difference

Assume:

(a) Total Acidity as H_2SO_4 = 87.31%
(b) Total Sulfuric as H_2SO_4 = 42.11
(c) Nitrosyl-sulfuric = 0.31

then the nitric acid as H_2SO_4 (d) is:

(d) = (a) − (b) = 87.31 − 42.11 = 45.20

and the total nitric acid as HNO_3 will be (e):

$$(e) = (d) \times \frac{HNO_3}{\frac{1}{2}H_2SO_4} = 45.20 \times \frac{63}{49} = 45.20 \times \frac{9}{7} = 58.08$$

Note: The equivalent weights of 63(HNO_3) & 49($\frac{1}{2}H_2SO_4$) are taken as close enough for ordinary works practice

In order to find the actual H_2SO_4 and the actual HNO_3 it is necessary to deduct from the above values the sulfuric and nitric acids equivalents in the nitrosyl-sulfuric acid

As the molecular weight of $HNOSO_4$ is 127 (approx), the HNO_3 equivalent will be:

$$0.31 \times \frac{63}{127} = 0.15\% \text{ and } 58.08 - 0.15 = 57.93\% \text{ actual } HNO_3$$

the H_2SO_4 equivalent will be:

$$0.31 \times \frac{98}{127} = 0.24\% \text{ and } 42.11 - 0.24 = 41.87\% \text{ actual } H_2SO_4$$

The results are reported as follows:

H_2SO_4	41.87%
HNO_3	57.93
$HNOSO_4$	0.31
H_2O	−0.11 (by difference)
Total	100.00%

Refs: 1) Hackh's Dict (1944), 376-R 2) G.D. Clift & B.T. Fedoroff, "A Manual for Explosives Laboratories", Lefax, Inc, Philadelphia, 4th ed (1944)

Gilles patented in England in 1883 the prepn of Nitro-molasses (See under Molasses)
Ref: Daniel (1902), 562–63 (Nitromélasse)

Gilsonite or Uintaite. A black, brittle, lustrous, bituminous, mineral consisting of solidified hydrocarbons. Found only in the US, namely in Utah and Colorado. Its sp gr is 1.05 to 1.10 (77°F); hardness (Mohs' scale) 2; penetration 0 (77°F); fusing point (B&R) 270–370°F. Behavior on heating in flame – softens and flows. It is soluble in carbon disulfide and mixes well with the fatty acid pitches in all proportions. Its numerous uses are listed in Ref 2, p 535-L

Accdg to Ellern (Ref 3, p 98), it is a versatile inexpensive fuel and somewhat of a binder and compaction aid

Refs: 1) Hackh's (1944), 377-L 2) CondChemDict (1961), 534-R & 535-L 3) H. Ellern, "Military and Civilian Pyrotechnics", Chemical Publg Co, NY (1968), 129 & 315 4) Kirk & Othmer, Vol 10(1966), pp 527–533 by H.O. Ervin 5) US Spec JAN-A-356A (Sept 1973)
Note: B&R means Ball & Ring Method

See Asphalt, Asphaltum or Mineral Pitch in Vol 1, p A496-L

Girard, Aimé of France prepd at the end of the 19th century in France the explosive "Nitrohydrocellulose" (See under Hydrocellulose in Vol 7)
Ref: Daniel (1902), 342 (Girard, Aimé); 380–81 (Hydrocellulose); 556–57 (Nitrohydrocellulose)

Girard, Charles of France patented, accdg to Daniel (Ref 1, p 342) and Giua (Ref 4, p 312), nine expl compns contg K picrate, castor oil, MNNaphthalene or charcoal and K perchlorate, or K chlorate, or K nitrate. Following are five typical formulations: a) K picrate 4, castor oil 7, MNN 14 & K perchlorate 75% b) K picrate 80, castor oil 10 & charcoal 6% c) K picrate 5, castor oil 6, MNN 9, K perchlorate 45 & K nitrate 35% d) K picrate 4, castor oil 10, K chlorate 80 & charcoal 6% and e) K picrate 4, castor oil 6, MNN 11 & K nitrate 79% (FrP 295671, 27 Dec 1899 – 12 April 1900)

The same chemist patented somewhat later (Ref 1, p 343) four expls contg K chlorate or perchlorate with "solidified oil" (huile solidifiée), which was prepd by adding to 100 parts of soap (completely dried at 120–130°), 10 to 15 parts of castor oil and then continuing to heat for 2–3 hrs

at 150–170°. After filtering the hot liquid it was poured into containers and kept at RT. When required it was heated to 80° and mixed with other ingredients. Giua (Ref 4, p 397), calls this substance "il grasso" and lists the following formulations: a) K chlorate 80 & grasso 20%
b) K chlorate 80, grasso 14 & MNN 6% and
c) K chlorate 80, grasso 14, Azobenzene 4 & K picrate 2% (FrP 295754, 30 Dec 1899 – 18 April 1900)

Four expl compns of Ch. Girard, patented in 1905, are listed on p 398 of Ref 4. They are mixtures of K chlorate with Azodinitrotoluene, with Azodiaminobenzene, with Picroazobenzene and with Azo-p-nitroaniline

An expl compn of Ch. Girard, patented in 1908 In the USA (Ref 2), was prepd by combining molten Trinitrocresol with PA in molecular proportion (to give a low melting compn), and while the mixture was still liquid some K chlorate was incorporated in the above mixture in order to neutralize the HCl formed during expln and to increase the power of expln

On p 290 of Ref 4are listed four low melting expl mixtures of PA with Nitronaphthalene, with DNT and two formulations with Trinitro-m-cresol patented by Girard in 1909 (Ref 3)
Refs: 1) Daniel (1902), 342–43 2) Ch. Girard, USP 894254 (1908) & CA **2**, 3283 (1908)
3) Ch. Girard, MonitScient **1909**, p 246 4) Giua, Trattato **6**(1)(1959), 290, 312, 396–97 & 398

Girard, Millot & Vogt studied during the Paris siege of 1870 by the Germans, various absorbents for NG in Dynamites. Among the available substances they found that lump sugar (sucre en morceaux) was a good absorber. They prepared a Dynamite consisting of sugar 60 & NG 40% and found that it was very insensitive to impact. No detonation was produced when a 4.7kg weight was dropped from a height of 1.65 meters
Ref: Daniel (1902), 343

Giua, Michele, Dr (? – 1973). Italian scientist. Professor of industrial organic chemistry at University of Torino (Turin). Specialist in expls and author of numerous publications. His book of 1959, entitled "Trattato di Chimica Industriale", Vol **6**, Part 1, published by UTET in Torino was devoted to explosives on pp 1 to 497 ("Esplosivi", in collaboration with Dr Maria Luisa Marchino); to Chemical Warfare Agents on pp 498 to 556 ("Aggresivi Chimici", in collaboration with Dr Marco Civera); and to Matches on pp 557 to 609 ("Fiammiveri" by Dr Leopoldo Stefanine)

Glacial Acetic Acid (AcOH). An anhydrous acetic acid (See Vol **1**, p A25-L) having an ice-like crystalline appearance (fp 16.7°)

Glaser of Berlin proposed in 1883 a proplnt prepd by treating the surface of NC grains with ether-alcohol soln, followed by evapn of solvent. This gave grains slower burning than untreated NC grains. In later patents (1887), the same inventor treated NC grains with other volatile solvents, such as et acetate, acetone, etc, and he also added to NC substances like nitrates, chlorates, picrates, naphthalene or paraffin
Ref: Daniel (1902), 344

Glass and Glass-Blowing. Glass is an amorphous substance, usually transparent or translucent and consisting of silicates, made by fusing together sand and alkalies (such as Na or K) and some base (such as Ca or Pb). Some glasses are borates or phosphates. Glasses may be considered as undercooled liquids of high viscosity. The art of glassmaking has been known for more than 4000 years, and it seems that the Egyptians were the first to produce it

In order to make articles of various shapes and sizes, glass is melted and then either blown or cast. The glass may also be pressed in the hot state. Venice, Italy has been particularly known for its skillful glass-blowers from the time of the Middle Ages. The art of glassblowing is of great importance to chemical and physical laboratories, and it is expeditious for a laboratory of any size to have expert glass blowers among its staff
Refs: 1) H. Vigreux, "Le soufflage de verre", Dunod, Paris (1931) (Good book by instructor at Sorbonne; used by senior author during his studies)

2) J.D. Heldman, "Techniques of Glass Manipulation in Scientific Research", Prentice-Hall, NY (1946) 3) A. Silverman, "Glass: Historical Notes 1900 to 1950", JChemEduc **30**, 32–4(1953) 4) E.W. Morey, "Properties of Glass", Reinhold, NY (1954) (ACS Monograph No 124) 5) L.M. Parr & C.A. Handley, "Laboratory Glass Blowing", ChemPublgCo, NY (1957) 6) A.J.B. Robertson et al, "Laboratory Glass Blowing for Scientists", Academic Press, NY (1957) 7) E. L. Wheeler, "Scientific Glass Blowing", Interscience, NY (1957) 8) W.E. Barr & V.J. Anhorn, "Scientific Glass Blowing and Laboratory Techniques", Instruments Publg Co, Pittsburgh (1959) 9) P. Beyersdorfer, "Glashuettenkunde", (Glassworking Science), VEB (Volkseigener Betrieb), Deutscher Verlag fuer Grundstoffindustrie, Leipzig (E. Germany) (1964) 10 Kirk & Othmer, 2nd Ed, Vol **10**(1966), pp 533–604 ("Glass") 11) J.O. Johnes, "Glass", 2nd Ed, Chapman-Hall, London (1971)

Glass in Priming Compositions. It is known that sensitivity of expl mixts to friction (percussion) is greatly increased when a small amt of ground glass [or of other abrasive material, such as sand or carborundum (See Vol **2**, p C65-L)] is mixed with the expl

A soft white soda-lime glass is most suitable for incorporation with expls like MF + $KClO_3$, etc

Requirements of the US Armed Forces are covered by Specification JAN-G-479 (July 1947) and are as follows:

1) *Composition* – soft soda-lime glass

2) *Workmanship.* The glass must be ground with French burrstones or other mills giving the shape, edge and other characteristics of burrstone grinding to the particles

3) *Foreign matter.* Material shall be free from foreign matter when examined under a low-power microscope

4) *Particle shape.* When examined under the microscope the fracture of the particles shall be conchoidal and there shall be no long needles or round particles

5) *Granulation.* When shaken for 5 minutes, either by hand or with a shaker geared to produce 300±15 gyrations and 150 10 taps per minute, the sample should comply with the requirements for class. The coarsest is *Class A:* thru No 60 US Std Sieve minimum 100%, retained on No 100 min 99% and the finest is *Class E:* thru No 170 min 90%, retained on No 200 min 75%

Glass Bulbs for Weighing Acids.

It is important that chemists working in acid laboratories know how to blow weighing glass bulbs and how to use them for taking samples of acids. This method is particularly suitable for sampling fuming acids (such as oleum or fuming nitric) because it avoids release of fumes

Following is the description given by Clift & Fedoroff, Vol 1 (1942), Chap 1, Part 2, pp 7–8 & 14:

THE ANALYSIS OF OLEUMS INCLUDING THOSE CONTAINING NITRIC ACID

Apparatus:

Nitrometer, preferably duPont (A.H. Thomas Co Cat)

Glass bulbs of about 15mm diameter

Preparation of Glass Bulbs:

Following is a brief description of the manipulation in making these bulbs: Take a piece of soft glass tubing about 5mm diam and heat to redness in a hot tapered flame. Remove from the flame and draw out a capillary about 1mm diam. Break off the end of the capillary and on the other end heat a small portion of the glass to redness (See a, Fig G16). Remove from the flame and blow a small bulb about 15mm diam (See b, Fig G16). A little practice will show how much glass to take for this purpose. It is important to have the capillary uniform and the walls of the bulb thick enough to stand subsequent heating

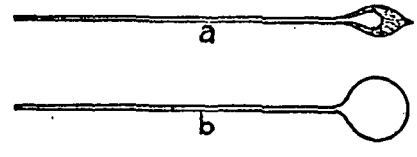


FIG G16

I. TOTAL ACIDITY

1) Weigh up two bulbs, one for a check, with the capillary stems about 7–8cm long

2) Shake the bottle well containing the sample, open it and cover the mouth with a piece of

light lead having a center hole of about 2–3mm in diam

3) Heat one of the bulbs gently in the flame of a Bunsen burner and quickly place it in the hole in the lead plate so that the end of the capillary is under the surface of the acid (Fig G17)

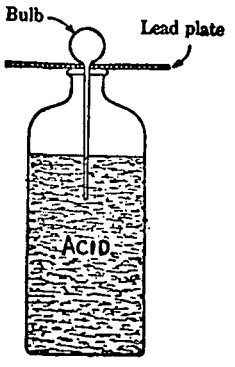


FIG G17

4) While the bulb cools and the acid fills it, prepare a heavy walled conical flask of 500ml capacity and place about 100–150ml of distilled water in it

5) Remove the bulb from the acid, carefully wipe the capillary dry and seal the end by means of the flame. By further heating, bend a hook on the end of the stem for hanging on the balance

6) Weigh the bulb containing the acid and drop it capillary first into the flask

7) Break the capillary, with a stirring rod, about 2cm from the bulb and allow the acid to drain into the water. In the case of strong oleums the action is liable to be violent and the bulb can be held lightly against the side of the flask with the stirring rod

8) Allow to drain until water has completely replaced the acid in the bulb and any fumes that have been absorbed. Then break the bulb under the water and crush the capillary into small particles

9) Add Methyl Red Indicator and titrate with sodium hydroxide solution until the color changes from red to greenish-yellow

Calculations:

$$\%\text{Total Acidity as } H_2SO_4 = \frac{(NF)\times 0.04904\times 100\times R}{W}$$

where: (NF) = Normal Factor of the NaOH soln
0.04904 = Equivalent weight of H_2SO_4 in 1 ml of Normal soln
R = Burette reading
W = Weight of sample taken

DuPont's nitrometer is described in Vol 1 of Encycl, pp A373 to A396 with Fig on p A374-L. Cleaning of glass parts of nitrometer is described here under "Glass Cleaning and Solvents Used". Preparation of nitrometer for standardization is described on pp A373-R to A374-R and standardization on pp A374-R to A376-R

Replace "Determination of AN by Nitrometer Method" on p A376-R by the following:

Nitric Acid in Oleums

10) Add about 7–8ml of 85% nitric-free sulfuric acid to a 25ml weighing bottle, stopper and weigh

11) Remove the stopper and add a sufficient quantity of oleum to produce 70 divisions of NO in nitrometer. Replace the stopper and weigh the bottle

12) Shake the contents gently taking care not to smear the stopper and pour the contents carefully into cup (a) of bulb E, shown in Fig on p A374-L

13) Draw the contents of (a) into E, as described on p A375-L, beginning with the words: "By lowering F and opening f & c draw the contents of cup (a) slowly into E, etc" up to the words: "Before proceeding with actual measuring of vol of NO evolved", on p A376-L, 12th line from the bottom, proceed as in 14)

14) After transferring the gas into the burette D, manipulate A & D until the meniscus in C is even with the top of pasted paper P (See Fig on p A374-L) and the meniscus in D is on the level with it. (Under no circumstances replace compensating tube C)

15) Use *Notes* on p A376-R

16) *Calculation:* $\%HNO_3$ in Oleum $= \frac{R \times 63.02}{100 \times W}$

where: R = Reading of buret D in ml and
W = Weight of oleum sample in grams

Glass Cleaning and Solvents Used. A brief description is given in Vol 3 of Encycl, p C329-L under "Cleaning Solutions for Laboratory Glassware". A rather complete description is given

in Lange (Ref 3) and in ChemRubberHdb (Ref 4)

"Cleaning of Glassware Used in Calibration of TNT Thermometers" is described in Vol **2**, pp C7-R & C8-L, under "Calibration of TNT Thermometers"

Cleaning of glass parts of DuPont's nitrometer is conducted in the following manner: Fill all the glassware with a mixt of concd sulfuric acid and K dichromate to which a little oleum has been added. Let stand overnight, drain, and thoroughly wash first with tap water and then with distd w. After this, rinse 3 times with acetone, first with acetone previously used for 2nd washing (1st washing is always discarded), 2nd with acetone previously used for 3rd washing and 3rd with fresh redistd acetone. Dry by air thoroughly dehydrated by bubbling it thru two Drexel wash bottles contg concd sulfuric acid and finally thru glass wool (to catch acid droplets mechanically entrained)

In JChemEduc (Ref 2), the following cleaning solvents are suggested:

Hydrochloric Acid for removing basic tars
Na Hydroxide for acidic tars
Benzene for hydrocarbon residues
Acetone for partially oxidized tars

For greasy films a mixture of 1 volume propyleneglycol, 1 vol chloroform and powdered Castile soap dissolved in the mixt is recommended. It functions better when slightly warmed

Refs: 1) Clift & Fedoroff, Vol **1** (1942), Chap 1, p 28 2) E.M. Doss, JChemEduc **24**, p 537 (Nov 1947) 3) Lange (1961), 1776 4) ChemRubHdb (1962), 3420

Glass Dynamite. A mining expl prepd by G. Mowbry in 1873 by impregnating glass, blown into fine flakes with NG. This expl extruded easier than corresponding Guhr Dynamite, because the glass did not absorb NG (as does guhr), but simply held it on the surface. Due to glass being harder than guhr, Glass Dynamite was easier to detonate and it had a slightly higher detonation velocity than Guhr Dynamite

Refs: 1) Daniel (1902), 438 2) Van Gelder & Schlatter (1927), 419–20

Glass-Feather Manometer. A device for measuring pressures, invented by Schaeffer and Treub (ZPhysikChem **81**, 308 (1913) and made in the USA by F.E. Donath, 22 Fourth St, Aspinwall, Penna

This manometer was successfully used by the late Dr E. Berl et al [IEC **10220** (1938)] in modified Will's Stability Test Apparatus

Glass Temperature. Same as Brittle Point or Brittleness Temperature described in Vol **2** of Encycl, pp B302-L to B303-L

Glauber, Johann Rudolph [1603 (or 1604)–1668]. Dutch (or German) "iatrochemist" [belonging to the 16th century school of medicine basedon principles of Swiss physician Paracelsus (1493–1541)] who prepd expl substances Potassium Picrate and Ammonium Nitrate. He also prepd the salt known as "Glauber salt" (cryst Na sulfate) and pure nitric and hydrochloric acid. He also worked with saltpeter and Gunpowder

Refs: 1) Hackh's Dict (1944), 378 (He gives year of birth as 1603 while Brieger gives 1604) 2) W. Brieger, SS **12**, 304–07 (1917), "Johann Rudolph Glauber als Sprengstoff-Chemiker"

Glazing or Graphiting of Propellants (Lissage ou Plombinage des Poudres, in Fr). In order to make a cannon propellant (either black or smokeless), so that it can be blended and poured into containers or cartridges without danger from static electricity, the grains are rendered smooth and glossy by rotating them together with a small amount of powdered graphite in a drum or in a "Sweetie Barrel", such as shown in Davis (Ref 3, p 291). Another purpose of this operation is to render the grains nonhygroscopic. Proplnts which are required to burn quickly (such as rifle and sporting proplnts) do not receive graphite treatment but are rendered smooth by rotating them longer than graphited proplnts

Refs: 1) Marshall **1** (1917), 83 2) Pepin Lehalleur (1935), 306 3) Davis (1943), 291 & 306 4) J. Fauveau & R. Delpy, MP **31**, 162 (1949)

Glide Bombing. See Vol **5**, p D1523-R under "Dive Bombing" and also in Ohart (1946), p 202

Gliding Torpedo GT-1. A 2000-lb aerial torpedo developed by the US Navy during WWII. It was released from a plane at a distance up to 25 miles from a surface target (such as ships concentrated in a harbor or in a convoy)
Ref: Anon, Ordnance **31**, 384 (1947)

Gliossiline or Abelite. Accdg to Giua (Ref) it was a Dynamite-type expl patented by F. Abel in 1867: NG 65.5, Collod Cotton 30.0, K nitrate 3.5 & Na carbonate 1.0%
(Compare with Abel Powder described in Vol **1**, p A1-R)
Ref: Giua, Trattato **6**(1)(1959), 342

Glmite. A mixture of liquid oxygen and finely pulverized carbon which was tried during WWI as a drop-bomb charge. The mixture had to be prepd just before flight because it was effective only for a short time after mixing
Refs: 1) T.L. Davis, ArmyOrdn **20**, 92 (1939) 2) Bebie (1943), 75

Globular Propellant Powder. An NC in EtAc lacquer is dispersed in an aqueous soln whose temperature is below the bp of EtAc. Thr suspension is then rapidly blended with 5X its vol of the same aqueous soln under superatmospheric pressure. The combination of temperature and pressure release causes the EtAc, and a little w, to instantaneously flash off, leaving spherical particles of the NC behind for centrifugal separation. The process is continuous
(Compare with BALL POWDER, described in Vol **2**, pp B11-R to B16-L)
Ref: R.L. Cook & E.A. Andrew (Assigned to Olin Mathieson Chem Corp), USPatent Office No 2888713 (2 June 1959), "Method of Preparing Globular Propellant Poqder" & CA **53**, 16541 (1959)

Glonoin. Name for pure NG used in medicine as 1% alcoholic soln, called *Spirit of Glonoin*, in heart diseases. The usual dose for angina pectoris is 1 drop of spirit taken in water. Glonoin is also administered in lactose or dextrose pellets, each contg 1/100 grain (0.0006g) NG, by dissolving them under the tongue
Ref: Davis (1943), 208

Gloves, Conductive. The expl industry has been looking for a long time for a glove that would provide al-in-one comfort, hand protection and safety against static electricity. A new electrically conductive glove made of "Velostat" is said to be the solution for use during static hazardous operations. The gloves drain off static charges when the wearer is grounded. Chemically resistant, they are claimed to provide protection from acids, alkalies, and other contaminants that can cause injury. Manufacturer especially recommends disposable gloves for those working with flammable solvents, powders, liquids, and dusts
Refs: 1) Velostat Gloves No 6090, Custom Materials, Inc, Alpha Industrial Park, Chelmsford, Mass, 01824 2) G. Cohn, Edit, Expls&Pyrots **4**(8), 1971
Note: "Velostat" is an acronym for very low static

GLTN. Abbr for Glycerolmonolactate Trinitrate

D-Glucitol (Sorbitol) and Derivatives
D-Glucitol (Sorbitol or Hexanhexol), $HOCH_2(CH_2OH)_4CH_2OH$; mw 182.17, ndls (with ½ or 1 w), mp about 110° (dry), 100° or less (with w), very hygr when dry; sol in w & hot alc. Found in various fruits; prepd by sodium amalgam reduction of d-sorbose or by pressure hydrogenation of dextrose with Ni catalyst. Used for prepn of ascorbic acid (Vitamin C), for synthesis of resins, surface-active agents, varnishes, syrups, cosmetic creams and for explosive Sorbitol Hexanitrate

Refs: 1) Beil **1**, 533, (281), [605] & {2385} 2) C. Vincent & Delachanel, CR **111**, 51(1892) 3) CondChemDict (1962), 1062-L (Sorbitol); (1971), 817-R

D-Glucitol (Sorbitol) Nitrate. Sorbitol was nitrated with cold mixed acid, an oil resulting. This exploded on hitting with a hammer. No analysis was reported
Refs: 1) Beil **1**, 534 2) C, Vincent & Delachanel, CR **108**, 355(1889)

D-Glucitol (Sorbitol) Hexanitrate, $O_2NOCH_2(CHONO_2)_4CH_2ONO_2$; mw 452.16, N 18.58%, OB to CO_2 +7.1%; tablets (from alc), mp 55.5°(Ref 1). Prepd in a mixed acid nitration at ambient temperature of an equiparts mixture of sorbitol and glycerol. The resulting oil was stable for 36 min at 80° in the Abel test, and jelled at −22°(Ref 2)
Refs: 1) Beil **1**, {2391} 2) P. Lhoste, MP **40**, 103–07(1958) & CA **55**, 3446(1961)

Glückauf. An older Ger permissible expl. Its Formulation AIII contd AN 82.7, DNB 1.0, woodmeal 11.5 & Cu oxalate 4.8%; its power by Trauzl Test was 341cc and charge limit 450g
Refs: 1) Marshall **1**(1917), 191 2) Clift & Fedoroff **2** (1943), p G2

d-α-Glucoheptose or d-Gluco-α-heptose, $HO.CH_2(CHOH)_5.CHO$; mw 210.18; colorless rhombic plts (from w) or prisms, mp 215°(dec); soly in w 10% at 20° and 47.6% at 100°; soly in boiling alc about 0.3%. May be prepd from the lactose of d-gluco-α-heptonic acid, described in Beil **18**, 235 & (419) as γ-Lacton der d-Gluco-α-heptonsäure (Ref 1)

On nitration it produces an expl compd:
d-α-Glucoheptose Hexanitrate, $O_2NO.CH_2(CH.ONO_2)_5.CHO$; mw 480.18; N 17.50%, OB to CO_2 +3.33%; colorless ndls (from alc); mp 100°; reduces Fehling's soln on warming. Can be prepd by nitrating d-α-Glucoheptose (Refs 2, 3 & 4). It is a powerful expl

Refs: 1) Beil **1**, 934 & (468) 2) Beil **31**, 360 3) W. Will & F. Lenze, Ber **31**, 68 (1898) 3) J. Leibowitz & S.H. Silman, Ber **58**, 1889(1925) 4) Davis (1943), 244

d-Gluconamide and Its Pentanitrate. Their properties are given in Vol **1**, pp A122-R & A123-L, under "Aldonic Acids, Their Derivatives and Nitric Esters", but prepn of pentanitrate is not given

Following procedure, which is a modification of Caesar's method (Ref 1), was used by Wolfrom et al (Ref 3):

The recrystallized d-gluconamide was slowly added, with stirring, to the chloroformic soln of nitrogen pentoxide (either alone or mixed with P_2O_5 or NaF) at −20°. Stirring was continued, occasionally, for the next 15 mins, while temp slowly rose to RT. After allowing to stand for 35–45 mins, the slurry was filtered and the solid portion was drowned (after allowing the chlf to evaporate) in ice water and transferred to a Büchner funnel. After rinsing ppt 3 times with water, under suction, it was dried in a vacuum over P_2O_5. This ppt was purified by dissolving in methanol and adding water to the cloud point. Melting point of dried product was 149°. It was more stable than d-gluconic acid pentoxide

Filbert (Ref 2) proposed using nitrated gluconamide in detonators and blasting caps
Refs: 1) G.V. Caesar et al, USP 2400287 (1946) & JACS **68**, 372(1946) 2) W.F. Filbert, USP 2444903(1948) & CA **43**, 1796 (1949) 3) M.L. Wolfrom et al (Ohio State Univ Res Foundation) Final Rept, Project **458** (Contract No DA-33-019-ORD-163, Project No TB 3-0110S), Columbus, Ohio (1952), p 2 & JACS **75**, 3662(1953)
Addnl Ref: H. Ficheroulle & A. Kavache, MP **41**, 14–18(1959)(Prepn and props)

d-Gluconic Acid and Its Pentanitrate are briefly described in Vol **1**, pp A122-R & A123-L under "Aldonic Acids, Their Derivatives and Nitric Esters". Gluconic Acid

Pentanitrate is less stable than Gluconamide Pentanitrate
Refs: Same as under Gluconamide and Its Pentanitrate

Glucopyranose and Derivatives
D-Glucopyranose, $HOCH_2CH(CHOH)_4O$; mw 180.16. Two forms are known, varying in the configuration of the –CHOH next to the ring oxygenation.
α-D-Glucopyranose crystallizes from w as monohydrated plates, mp 83–86°, dehydrating below 100°; crystallization from alc, methanol, or 30–35° w (satd) gives water-free needles, mp 146°. The dry form has sp gr 1.544 at 25°. At 20° 2g of the dry form will dissolve in 80% alc (100cc) and 0.85g will dissolve in methanol (100cc). It may be prepd from D-glucose by dissolving the dry form of the latter in w and diluting with cold HAc
β-D-Glucopyranose, crystallizes from alc, mp 146–150°, sp gr 1.547 at 13°; sol in 80% alc at 20° of 4.9g/100cc. It may be prepd from β-D-glucose by dissolving the latter in w, diluting with hot HAc, redissolving the precipitate in 0° w, and repptg with alc
Refs: 1) Beil **31**, 86, 87 2) C.S. Hudson & J.K. Dale, JACS **39**, 323–4(1917)

α-D-Glucopyranose Pentanitrate, $O_2NOCH_2CH(CHONO_2)_4O$; mw 405.16, N 17.28%, OB to CO_2 +2.0%; crysts, mp 53.5°, expl 250°, sp gr 1.75 at 20°; very sol in eth & methanol; sol in alc. Prepd by nitration below 0° of the parent in HAc/Ac_2O. Loses 60% of weight in 10 hours at 100°
Refs: 1) Beil, not found 2) G. Fleury & L. Brissaud, CR **222**, 1051–3(1946) & CA **40**, 5022(1946) 3) G. Fleury et al, MP **31**, 107–20(1949) & CA **46**, 11685(1952)

β-D-Glucopyranose Pentanitrate, $O_2NOCH_2CH(CHONO_2)_4O$; mw 405.16, N 17.28%, OB to CO_2 +2.0%; crysts, mp 111°, expl 257°, sp gr 1.78 at 20°; sl sol in alc, eth & methanol. Prepd by same method as α-isomer above. Loses 60% of weight in 6 hours at 100°
Ref: See α-isomer above

Glucosan and Derivatives
β-Glucosan or Levoglucosan (Anhydroglucose), $O.C(CHOH)_3CH.CH_2.O$, mw 162.14; rh crysts, mp 178°; sol in w & 90% alc; sl sol in alc. Prepd by heating barium hydroxide soln with p-acetophenyl-β-d-glucopyranoside at 100° for 4 hours
Refs: 1) Beil **1**, 894 & (452) 2) M. Tanret, BullSocChemFr [3] **11**, 950(1894)

β-Glucosan-2,3,4-trinitrate or **Levoglucosan-2,3,4-trinitrate**, $O.C(CHONO_2)_3CH.CH_2.O$; mw 297.14, N 14.14% OB to CO_2 –24.2%; ndls (alc), mp 101°(94–5°, Ref 4). Prepd by the mixed acid nitration of levoglucosan
Refs: 1) Beil **1**, 894 2) W. Will & F. Lenze, Ber **31**, 87(1898) 3) L.P. Kuhn, JACS **68**, 1761–2(1946) & CA **40**, 7161(1946) 4) A.J. Mian et al, JOC **27**, 1895–6(1962) & CA **57**, 7355(1962)
Note: According to E.E. Sancho, "Química de los Explosivos", Madrid (1941), p 179, this trinitrate is more powerful than Hexanitroglucose

GLUCOSE AND DERIVATIVES

d-Glucose *(Dextrose or Grape Sugar)*, $C_5H_{11}O_5.CHO$; mw 180.16; rhombic ndls (from alc), sp gr 1.544 at 25/4°; mp – loses 1 H_2O at 118–20° and then melts at 146–47°; sol in w & alcl insol in eth. Can be prepd by the action of HCl on starchy materials, causing incomplete hydrolysis. Used as a reagent for detecting CO in blood; in manuf of confectionary, production of alcoholic liquors etc

Gives on nitration explosive pentanitrate
Refs: 1) Beil **1**, 879 & (443) 2) Hackh's Dict (1944), 379-R & 380-L

d-Glucose Pentanitrate or **Nitroglucose**, $C_6H_7O(ONO_2)_5$; mw 405.16, N 17.29%(theory); N 16.96% (found); viscous syrup which hardens at about 0°; mp <10°(Ref 2); bp – decomp at 135°; insol in w & in ligroin; very sol in alc. Can be prepd by nitrating d-glucose with mixed nitric-sulfuric acid (Refs 1, 2 & 3)

It is an expl which is unstable above 50°

Sancho (Ref 3) lists the following mixture used in Spain: Nitroglucose 10, AN 80, K chlorate 5 & coal tar 5%

Refs: 1) Beil **1**, 897; **31**, 146 2) W. Will & F. Lenze, Ber **31**, 74(1898) 3) Sancho (1941), 175 4) Davis (1943), 241 5) L. Brissaud, MémServicesChimÉtat **30**, 120–32(1943) & CA **41**, 715(1947) 6) M.L. Wolfrom et al, JACS **73**, 874–5(1951) & CA **45**, 5622(1951)

Glucose for Preparation of Sorbitol

Due to large amounts of *sorbitol*, $HOCH_2(CHOH)_4.CH_2OH$, being required during WWII for the prepn of ascorbic acid (Vitamin C), a method was developed by Tettamanzi & Arnaldi (Ref) for the prepn of sorbitol by the hydrogenation of glucose. Their method required smaller amts of catalyst, lower temperatures, lower pressures and shorter time than previously described methods

[See also D-Glucitol (Sorbitol) and Derivatives]

Ref: A. Tettamanzi & N. Arnaldi, AttiAccadSciTorino **77**, 271–77(1942) (An abbreviated transln is available at PicArsn Library)

Glucose-Glycerol and Lactode-Glycerol Nitrates. Solutions of glycerin with either grape sugar (glucose) or milk sugar (lactose) produce on nitration compounds similar to nitrated glycerin-sucrose mixtures, suitable for use in low-freezing dynamites

Glucose dissolves in glycerin more readily than sucrose and sucrose more readily than lactose

Glucose dissolves in glycerin to the extent of about 40 parts in 60, but for the reason of stability it is better to use a mixture of 20p of glucose and 80p of glycerin. Such a mixture yields on nitration an oily compound with N=18.20% and consisting of Glucose Pentanitrate and NG. This mixture does not differ appreciably in expl strength from that of straight NG

Lactose-glycerin solution on nitration yields a mixture of lactose octanitrate, $C_{12}H_{14}O_3(ONO_2)_5$, and NG

Ref: Naoúm, NG (1928), 256

Glucosides and Glycosides. *Glucosides* are compds of glucose with some other substances. There are methyl-, ethyl-, etc ethers of glucose, which occur in many plants. They belong to *glycosides*, which are compdx of sugars with other substances. Compds of fructose are known as *fructosides* and of galactose are *galactosides*. Glucosides treated either by heat, dilute acid, enzymes, bacteria, or fungi, yield glucose, while fructosides yield fructose and galactosides give galactose. There are alpha and beta glucosides

Refs: 1) Hackh's Dict (1944), 380-L (Glucoside); 384-L (Glycoside) 2) CondChemDict (1971), 423(Glycoside)

Glucosides, Nitration Studies were conducted during WWII by M.L. Wolfrom and described in OSRD **147** or PBA 31201(1941), entitled "The Preparation of Sugar Alcohols and Glucosides for Nitration Studies"

An example of glucosides is:

α-Methyl-d-glucopyranoside Tetranitrate, $O_2NOCH_2CHO[CH(ONO_2)]_3CHOCH_3$; mw 374.18, N 14.97%, OB to CO_2 –21.4%; plates (alc), mp 49–50°, dec 135°. Prepd by dissolving α-methylglucoside in cold nitric acid(sp gr 1.52), followed by dropwise addition of cold concd sulfuric acid at 0°

Refs: 1) Beil **31**, 182 2) W. Will & F. Lenze, Ber **31**, 80(1898) 3) L. Brissaud et al, MP **33**, 187(1951) 3a) L. Médard, MP **33**, 193–98(1951) 4) D. O'Meara & D.M. Shepherd, JCS **1955**, 4232–35 & CA **50**, 10655(1956) (They prepd several nitrated derivs of glucoside, but only the tetranitrate was expl)

Glucoside Nitrate in Mixture of Nitrated Polyethyleneglycol was described by R.C. Moran in CanadP 275876(1927) & CA **22**, 1687(1928)

Glues. See also under "Gelatin and Glue", this Vol, p G43-R & G44-L

Animal (Colla): Impure gelatinous matter of animal origin, most commonly *bone glue* made from hides and bones, *casein glue* made from skimmed milk, and *fish glue* made from fish skins[Hackh's

Dict (1944), 380]

Vegetable: A gelatinous mass obtd from different plants, the best being *Acacia glue* or *Gum arabic* prrpd from gummy exudations of the Acacia senegal. Glue known as *albumen* may be prepd from flour in starch manufacture. There are also glues prepd from agar-agar, dextrin, starch, potato flour, etc. Some are used in the expl industry, either as bonding agents or to increase the plasticity of expls. For instance, dextrin and gum arabic are used in some primer mixtures

Dextrin, starch, potato flour and the so-called gum sugar which is a syrupy, sticky and non-crystallizing soln of sugar in glycerin were patented by Dynamit AG of Germany for increasing the plasticity of dynamites inexpensively [Naoúm, NG (1928), p 406]. Same author also cites on p 351 a method of prepn of Gelatin Dynamite in which glue was used [GerP 172651(1905) issued to Schachtebeck]

Glue-Gelatines. Compounds such as "Melan" (See under) were used for the prepn of plastic dynamites, partly replacing gelatinized NG

Glue, Nitrated. Daniel (1902), p 520) describes one of the methods of nitration of glues. For this he advises heating the glue with a small amount of dil nitric acid until dissolved, then gradually adding mixed acid with stirring; the product is washed and dried

Glues Used for Military Purposes (US Specifications). Some of the adhesives, binders & glues used for military purposes are as follows:

Adhesive and Sealing Compounds, Cellulose Nitrate Base, Solvent Type (For Ordnance Use); US Spec MIL-A-82484 (June 1967)

Adhesive MR-23 (For Use in Ammunition); US Spec MIL-A- 50926(1) (April 1972)

Adhesive, Dextrin (Spiral Tube Winding for Ammunition Containers); US Spec MIL-A-13374C (June 1969)

Adhesive Paste, For Demolition Charges; US Spec MIL-A-374A (Jan 1953)

Adhesive, Phenol and Resorcinol Resin Base (For Marine Service Use); US Spec MIL-A-22397 (Sept 1960)

Adhesive, Silicone Rubber for Igniter Mark 265; US Spec MIL-A-23940 (Sept 1966)

Adhesive, Water-Resistant (For Sealing Fiberboard Boxes); US Spec MMM-A-250A (April 1967) (For Sealing Waterproof Paper); US Spec MMM-A-260A (Sept 1966)

Binder, Cellulose Nitrate (For Pyrotechnic Mixtures); US Spec MIL-B-1085A (Jan 1951)

Glue, Animal (Protective Colloid); US Spec MIL-G-40630C (Oct 1972)

Glue, Marine, and Aviation Marine (Waterproof); MIL-G-413B (Oct 1963)

Gum, Arabic (Gum Preservative or Gum Acacia); US Spec JJJ-G-821. Used as an adhesive, bonding agent, fuel and retardant in pyrotechnics Source & props are given in AMCP **706-187** (Oct 1963)

Gum Tragacanth (For Use in Ammunition); US Spec JAN-G-96A (Aug 1969). Used as a bonding agent & fuel in pyrotechnics. Source & props are given in AMCP **706-187** (Oct 1963)

See also **Adhesives** in Vol **1**, p A102-R; **Binder or Agglutinant** in Vol **2**, p B120-R; and **Bonding Agents or Adhesives for Ordnance** in Vol **2**, p B242-L

Glug & Slug-Units of Mass

Concern was expressed by David (Ref 2) about the possibility that a new unit of mass, *glug*, suggested by Price (Ref 1), might prove confusing, but, in the opinion of Dunkle (Ref 3), it should not be. As long as we have *slug*, why not the *glug?*

If we let g represent unit acceleration, the new unit can be expressed as:

$$1\ \text{glug} = 1\,\frac{\text{gram-force}}{g} = \frac{981}{1} \cdot \frac{\text{dyne}}{\text{cm/sec}^2} = 981\ \text{grams-mass},$$

just as in English system:

$$1\ \text{slug} = 1\,\frac{\text{pound-force}}{g} = \frac{32.2}{1} \cdot \frac{32.2\ \text{poundals}}{\text{ft/sec}^2} = 32.2\ \text{pounds-mass}$$

Refs: 1) E.W. Price, AmJPhys **25**, 120 (1957) 2) A. David, Ibid **26**, 41(1958) 3) C.G. Dunkle, Ibid **26**, 503(1958)

Glukodine. Accdg to Daniel (Ref 1), it was a liquid expl prepd at the end of the 19th century in Sweden by C.G. Björkmann by nitrating a sa-

turated soln of sugar in glycerine. This mixture was proposed by Dittmar to be used in mining expls: a) Glykodine 36.40 (NG 33.19 & NSugar 3.21), sugar 8.40, Na nitrate & chlorate 31.20 & NC 23.36 parts
b) Glykodine (NG 30.23 & NSu 4.03), sugar 8.76, Na nitrate & chlorate 37.84 & charcoal 19.31 parts. These expls are called by Sancho (Ref 2) "Explosivos de Björkmann"
(Compare with "Björkmann Explosive", described in Vol **2** of Encycl, p B165-L)
Refs: 1) Daniel (1902), 345 2) Sancho (1941), 181–82

Glutaric Acid and Derivatives
Glutaric or Pentanedioic Acid (n-Pyrotartaric Acid), $COOH(CH_2)_3COOH$; mw 132.11, colorless crysts, sp gr 1.429 at 15°, mp 97.50, bp 200° at 20mm; sol in w and in benz; ver sol in alc and in eth. Can be prepd from cyclopentanone, $CH_2(CH_2)_3CO$. Used in org synthesis
Refs: 1) Beil **2**, 631, (272) & [564] 2) CondChemDict (1961), 539-R; (1971), 420-L
Glutaric Acid Diazide, $H_2C(CH_2CON_3)_2$; mw 182.14, N 46.15%; clear, pungent fluid oil, explodes on heating even under w. Prepd from the dihydrazide hydrochloride salt in eth with sodium nitrite
Refs: 1) Beil **2**, 635 2) T. Curtius & H. Clemm, JPrChem [2] **62**, 196 (1900)

Glutaricacidglycine and Derivatives
Glutaricacidglycine or Glutarylglycine, $HOOC(CH_2)_3CONHCH_2COOH$; mw 189.17. There is no reference to this compound in the open literature
Refs: 1) Beil, not found 2) CA, not found
Azidoglutaricacidglycine Azide, $N_3CO(CH_2)_3CONHCH_2CON_3$; mw 239.20, N 41.00%; thick oil, explodes in a flame. Prepd from the corresponding dihydrazide and nitrous acid at 0°
Refs: 1) Beil **4**, [791] 2) T. Curtius & W. Hechtenberg, JPraktChem [2] **105**, 324 (1923)

Glutazine and Derivatives
Glutazine, 4-Amino-2,6-dihydroxypyridine, or *4-Imino-2,6-dioxopiperidine,* $H_2N.C_5H_2N(OH)_2$ or $HN:C_5H_5N(:O)_2$; mw 126.12, plates (from w), mp 300° (dec); sl sol in alc & hot w; insol in usual solvents. Prepd by boiling with soda soln β-hydroxy-β-amino-glutaric acid ethyl ester amide, or by heating at 130° 2,4,6-trihydroxypyridine and ammonium acetate
Refs: 1) Beil **22**, 511 & (656) 2) H.N. Stokes & H. von Pechmann, Ber **19**, 2696, 2705 (1886)

Azidoglutazine. There is no reference in the open literature to a compd corresponding to a mono- or a diazidoglutazine
Refs: 1) Beil, not found 2) CA, not found

Nitroglutazine or **3-Nitro-4-amino-2,6-dihydroxypyridine,** mw 171.11, N 24.56%; orn-yel plates (from w), dec 170–80°. Prepd from glutazine and nitrous gases in cold w
Refs: 1) Beil **22**, 512 2) H. von Pechmann, Ber **20**, 2656 (1887)

Dinitroglutazine or **3,5-Dinitro-4-amino-2,6-dihydroxypyridine,** mw 216.11, N 25.93%; OB to CO_2 −44.4%; yel plates (from w), dec on heating. Prepd from glutazine and nitrous gases in cold w
Refs: 1) Beil **22**, 512 2) H. von Pechmann, Ber **20**, 2656 (1887)

Gluten. A yellowish to gray pdr, or a gray brown sticky tough mass that is insol in w, sol in alkali and in strong AcOH. It is a mixt of proteins usually derived from corn or wheat, but applicable to similar material from other grains, such as rye, oats, etc. Gluten is the protein present in flour and bread. Used in certain breakfast foods, for adhesives, for prepn of amino acids and as an ingredient (in lieu of cereal meals) of some mining expls, such as Ammondynamits
Ref: CondChemDict (1961), 540-L; (1971), 420-L

Glutol or Glutoform. See under Formaldehyde-Starch Mixtures, this Vol, p F167

Glyceraldehyde (dl) or **Glyceric Aldehyde (dl),** (also called *Glyceryl Aldehyde*), $HO.H_2C.CHOH.CHO$; mw 90.08; colorless, tasteless crysts (from alc-eth mixt), sp gr 1.455 at

18/18°, mp 142°(145°), bp 150° at 0.8mm; sl sol in w, alc or eth; insol in benz, petr eth or pentane. It is produced by oxidation of sugars. Used in adhesives, as cellulose modifier, leather tanning and in prepn of polyesters
Refs: 1) Beil 1, 845, (427) & [888] 2) CondChemDict (1961), 540-L .(1971), 420-R

Glyceride. An ester of glycerol and fatty acids in which one or more of the hydroxyl groups of the glycerol have been replaced by acid radicals. Glycerides occur in nature and can be made synthetically. The most common ones are based on fatty acids and occur in oild and fats. Mono- and triglycerides are of commercial importance
(See under Glyceryls)
Ref: CondChemDict (1961), 540-L & R; (1971), 420-R

Glycerin(e). Same as Glycerol or Glyceryl Trihydroxide. Not to be confused with *glyceryl* or *propenyl*, which is trivalent radical, $-H_2C.CH.CH_2-$, derived from glycerol

Glycero. Same as Glyceryl

Glycerogen. A colorless, viscous glycerin-like liquid developed in Germany during WWII as a substitute for glycerol which was then in short supply. It was produced commercially by IGFarbenindustrie AG. The process was described in detail by Sheely (Ref 1). The mixt produced by IGFarbenind by continuous catalytic hydrogenolysis of sugar at 200° & 325 atms consisted of 35% glycerol, 35% glycols and 25–35% of hexitols (hexols) with some other compds. It was used in cellulose films, sausage casings, printing pastes, pharmaceuticals, etc and its nitrated product was used in lieu of NG in Dynamites (Refs 1 & 3)

Acodg to Ref 2, in the German process a 70% aqueous soln of sugar, contg 0.1% oxalic acid and 5 parts of Ni catalyst per 100p of dry sugar, was heated at 70° for 4–5 hours. The resulting inverted sugar was hydrogenated at 200° and 300–325 atm, which yielded a mixt of 40% glycerol, 40% glycols (mostly propylene glycol) and 20% of nonvolatile hexols (C_6 alcohols). On nitration this mixture gave an expl nitrate which was too unstable for military purposes, but suitable as an ingredient of Dynamite and other commercial expls

Refs: 1) M.I. Sheely, "Glycerogen, a Substitute for Glycerin", BIOS Miscellaneous Report No 23(1948) 2) Anon, JChemEduc **25**, 288(1948) 3) Fedoroff et al, PATR **2510** (1958), p Ger 70-L

GLYCEROL OR GLYCERIN AND DERIVATIVES

Glycerol or Glycerin (Glycyl Alcohol, Propenyl Alcohol or Glyceryl Trihydroxide)(Glycérine in Fr, Glyzerin or Glycerin in Ger, Glicerina in Ital or Span, Glitserin in Russ, and Gurizerin in Japan), $CH(OH).(CH_2OH)_2$; mw 92.09, sp gr 1.260 at 20/4° and 1.265 at 15/15°, mp 17.9°, fr p 0°(with supercooling), bp 290°. First isolated by Scheele in 1783, glycerol is a sweet tasting, extremely hygroscopic liq; sol in w and in alc; insol in benz, chlf, CS_2, CCl_4, eth, petr eth, and oils; the heat of combustion is 397.2kcal/mole

Preparation

There are several methods for the preperation of glycerol:

1) Recovery from the spent lye liquor obtd on *saponification* of fats and oils in the soap industry

2) Fermentation of sugar process developed in Germany during WWI, giving the so-called "Protol" glycerin

3) Preparation from propyl alcohol developed during WWII by IGFarbenindustrie at Oppau, Germany (the so-called "Oppau Process")

4) The Shell Chemical Corp synthetic method (Ref 5) from cracked gases containing propylene, which goes thru the following stages:

$$\underset{\text{Propylene}}{CH_3.CH{:}CH_2} \xrightarrow[500°]{Cl_2} \underset{\text{1,2-dichloropropane}}{CH_3.CHCl.CH_2Cl} \xrightarrow{\text{heat}}$$

$$\underset{\text{allyl chloride}}{CH_2{:}CH.CH_2Cl} \xrightarrow[20°]{Cl_2} \underset{\text{1,2,3-trichloropropane}}{CH_2Cl.CHCl.CH_2Cl} \xrightarrow{NaOH}$$

$$\underset{\text{glycerin}}{CH_2OH.CH(OH).CH_2OH}$$

It is also possible to treat allyl chloride with HOCl, transforming it to glycerin dichlorohydrin which, on action of NaOH, yields crude dilute glycerol consisting of a mixture of glycerol and NaCl in water. Final steps in the process are those of concentration, desalting and purification

The major use for glycerol is to form esters with phthalic anhydride to make alkyd resins; other major uses are in cellophane, drugs and toilet goods, tobacco, and, much less now that EGDN use has increased, in NG (requiring a 99% purity grade called **"dynamite glycerin"**)

Note: Acvording to Marshall (Ref 2), glycerol prepd by fermentation of sweet liquors might contain some trimethylene glycol, $CH_2.(CH_2OH)_2$, which being of lower density (1.055 at 20/20°) will lower the density of the glycerol. This impurity is not harmful as it gives a stable expl dinitrate on nitration. However, as the latter is more sol than NG and is more unstable in contact with acids, lower yields are obtained

General Refs on Glycerol:

1) Beil **1**, 502, (266), [575] & {2297}
2) Marshall **1** (1917), 201; **2** (1917), 703–706; **3** (1932), 41 & 229 3) J.W. Lawrie, "Glycerol and Glycols", Reinhold, NY (1928) (ACS Monograph No 44) 3a) Naoúm, NG (1928), 30–40 4) V. Grignard, Ed, "Traité de Chimie Organique", Vol **6** (1940), pp 381–545, Masson, Paris (R. Delaby, "Triols et Dérivés")
5) C.S. Miner & N.N. Dalton, Eds, "Glycerol", Reinhold, NY (1953) (ACS Monograph No 117)
6) C. Lüttgen, Ed, "Glyzerin und Glyzerinähnliche Stoffe", 2nd Edn, Strassenbau, Chemie und Technik Verlagsgesellschaft mbh, Heidelberg (1955) 7) Kirk & Othmer **10** (1966), pp 619–631, 17 refs (J.C. Kem, "Glycerol")

Glycerol Analysis

In testing glycerol, it should be remembered that it is very hygroscopic and has to be kept in well-stoppered containers

The requirements of the US Armed Forces for *"Dynamite Glycerin"* or *"High-gravity Glycerin"* (also known as Grade B) are coveredby Federal Specification O-G-491 and are as follows:

1) *Appearance.* It shall be clear and free from suspended matter when examined by transmitted light

2) *Color.* Maximum limit shall not be darker than a combination of No 80 yellow Lovibond glass and No 8 red Lovibond glass with a 5¼ inch column of glycerol. The test may be made by placing the sample in a colorimeter tube to a depth of 5¼ inches and comparing the color with an empty tube plus the two Lovibond glasses placed above or below the tube

3) *Odor.* Shall be slight and characteristic of the grade of glycerin specified. Compare with the sample agreed upon

4) *Specific Gravity.* Shall be not less than 1.2620 at 15.5/15.5°. Any method that is accurate within two points in the 4th decimal place may be used. The most convenient is the pycnometer method (See Vol **3** of Encycl, p D69-L)

5) *Acidity or Alkalinity.* When 50ml of glycerol is mixed with 100ml of distd water which has been freshly boiled and cooled (CO_2-free), and about 0.5ml of phenolphthalein indicator (5g dissolved in 1 liter of 50% alcohol), the soln shall not require more than 0.30ml of normal HCl or more than 0.30ml of normal NaOH soln for neutralization. It is more convenient to use approx 0.5N solns and multiply the results by the normality

6) *Ash.* Shall not exceed 0.01%. For this test, a 50g sample is weighed on a triple-beam balance into a dish, previously tared on an analytical balance, and heated over a free flame until the vapors continue to burn after removing the flame. (During the operation the sample should be protected from drafts). When the combustion dies out, ignite the residue at low red heat (or in a muffle furnace at 800°) until the carbonaceous matter is consumed, then cool in a desiccator & weigh

$$\% \text{ Ash} = \frac{\text{Wt of residue} \times 100}{\text{Wt of sample}}$$

7) *Chlorides, calculated as chlorine.* Shall not exceed 0.01%. For this test, add about 25ml of hot distd water to the ash residue and rub with the flattened end of a stirring rod. Add 1 ml of K chromate indicator (10g in 100ml water) and titrate with 0.01N Ag nitrate soln (1.7g pure $AgNO_3$ in 1 liter water) to the first

permanent reddish tint. Run a blank detn on the reagent and make the correction

$$\% \text{ Chlorides as } Cl_2 = \frac{\text{ml } AgNO_3 \times N \times 3.546}{\text{Wt of sample}}$$

Besides the tests described, the following tests may be used if desired:

8) *Moisture.* As water is strongly retained by glycerol, it is difficult to obtn correct results glycerol dehydration methods: drying a thin film of glycerol over concd sulfuric acid in a vacuum desiccator or by heating the film at 90° on a water bath to constant weight, as described in Naoúm (Ref 1, p 32), or by a method described by Laurie (Ref 2, p 259). More reliable results are obtd by using Carl Fischer's method or by distg glycerol with tetrachlorethane as proposed by T. Berth (Ref 3) and modified by Riesener & Kessen (Ref 4) (See Vol 5 of Encycl, pp D1620-L to D1628-L). Moisture may be calculated approx after determining the sp gr of the sample (See Table under tests by Hercules methods)

Following are the tests used by Hercules Powder Co, Wilminton, Delaware:

1) *Color:* Note the color and report in terms of "light straw", "straw", or "dark straw"

2) *Odor:* If it does not smell bad, report "no bad odor", otherwise describe the odor present

3) *Specific Gravity and Moisture Content:* Determine the sp gr by means of a 50ml pycnometer at exactly 15.6/15.6° or determine it at other temps and calculate for 15.6/15.6° as follows:

$$\text{Sp Gr at } 15.6/15/6° = \frac{G}{C} \times \frac{1}{1+a(t°-15.6°)} + B(t°-15.6°)$$

where: G = Grams of glycerol at t°

C = Capacity of pycnometer in grams of water at 15.6°

a = Coefficient of expansion of glass, 0.000025 per 1°C

t = Observed temperature, °C

B = Change in sp gr per 1°C:

0.00061 between 15.6 and 20°;
0.000615 " 20 and 25°;
0.00062 " 25 and 30°

Note: The advantage of making the sp gr detn at t° (such as RT) rather than at 15.6° is that the bath can be kept at a constant temp more easily at somewhat higher temps. Moreover, if the detn were made at 15.6°, the moisture condensing from the air on the surface of the pycnometer would not allow its being weighed until it assumed RT

Bosart and Snoddy (Refs 5 & 6) give a table of specific gravity vs % glycerol content for aqueous solns. A more detailed table is given in Hercules Manual (Ref 7), which is not reproduced here

4) *Acidity or Alkalinity.* The test is similar to the one described above (taken from Federal Specification O-G-49)

Note: The presence of fatty acids may be determined as follows: Add to a 250ml beaker about 200ml of water and place a pin-head size grain of camphor on the surface. (The beaker should be perfectly clean (greaseless) and should not be touched on the inside with the fingers). If the water is free of grease, the camphor will start to move in a rotary manner. Add a few ml of the glycerol to be tested and observe the behavior of the camphor. If it stops and remains motionless, it indicates the presence of fatty acids. Mineral oils are without effect

5) *Ash.* Same test as in Spec O-G-49, described above

6) *Chlorides as Chlorine.* Same test as in Spec O-G-49, described above

Note: As chlorides such as NaCl are volatile, part of them may be lost during the heating of carbonaceous residue either with a burner or in a muffle furnace, as in the test of Spec O-G-49. More accurate results may be obtd if the burning is carried only to the char and not to the ash. In this case proceed as follows: Over a Bunsen burner, heat a tared porcelain or platinum dish contg a 50g sample of glycerol until the vapors ignite after withdrawal of the flame. Allow the combustion to proceed without further heating until the flame dies out. Cool the dish, add 25ml of water and heat with stirring to almost boiling, then filter thru No 2 Whatman paper. Cool the soln to RT and titrate to the first permanent red color with 0.01N Ag nitrate in the presence of five drops of saturated K chromate indicator

Authors' Note: It is much easier to obtn a correct endpoint if the titration is carried to

a distinct red color and then back titrated to a yellow color by using 0.01N KCl soln
7) *Water.* Karl Fischer Method is used (See Vol 5 of Encycl, pp D1622-L to D1628-L)
Nitration Test: The "dynamite glycerin" might pass all the chemical and physical tests and still possess properties undesirable in the manuf of NG, such as slow separation from mixed acid, formation of emulsions and poor settling during washing, poor yields etc. For this reason a trial laboratory nitration of glycerol with exact detn of NG is of great value. The compn of mixed acid and the method of nitration should follow plant practice as closely as possible

When detn of yield is requircd, use a large sample of glycerol (about 100g) and Method No 1, which is conducted in an open lead casserole of about 1 liter capacity using for an agitator a thermometer inserted in a lead pipe with a slot for reading

Nitration can also be done in a glass nitrating funnel as described by Naoúm (Ref 1, p 34)

Method 1 (Laboratory)

Procedure:

The nitration must be conducted under a hood and behind safety glass. Avoid smelling of acids of resulting NG

1) Place the nitrator (lead casserole of 1 liter capacity) into a large earthenware dish contg a cooling mixture of ice and large crystals of common salt
2) Charge the nitrator with 600g of acid contg 49–50% HNO_3 and 52 to 53.5% H_2SO_4 and cool it to about 10°(50°F), using the armored thermometerfor stirring
3) Weigh into a small tared separatory funnel about 100g of glycerol
4) Attach the funnel above the nitrator in such a manner that the tip of the tube is a few inches above the surface of the acid
5) Open the stopcock of the funnel with one hand and allow the glycerin to flow in a small stream into the acid
6) Continue to agitate the acid by means of the armored thermometer held in the other hand
7) Watch the temp closely and, as soon as it approaches 15°, reduce the flow of glycerol in order not to exceed 15.5°(60°F)
8) By regulating the flow of glycerol, try to keep the temp range between 13 & 15°
Note: If the temp rises above 15°, stop adding the glycerol. If the rise in temp is fast with evolution of copious brownish-red fumes, drown the mixture in at least 5 liters of water in a beaker or casserole. Never dump into the sink
9) After all the glycerol has been added, continue the agitation for about two mins
10) Remove the nitrator from the cooling dish and transfer the acid-NG mixt to a 1000ml separatory funnel
11) Wash the nitrator with a small amount of fresh mixed acid and transfer the washings to the funnel
12) Let the mixt stand for 30 mins and run the acid layer into a 500ml separatory funnel
13) Leave the NG in the large separatory funnel and slowly add to it about 100ml of cold distd water
14) Insert thru the top opening of the funnel a small glass tube connected to the compressed air system and agitate the mixt, water-NG, for 5 mins
15) Allow to settle and run the NG into a small flask and the acid water into a larger one
16) Transfer the NG to the above 1000ml funnel and repeat the operation of washing, agitation and separation twice. The temp of the water should be about 30° for the second and about 40° for the third wash
17) Next wash with 100ml of about 3% aqueous Na_2CO_3 at 40°, agitate for 10 mins, separate and finally wash with 100ml of concd NaCl soln at 40°
Note: As NG is appreciably soluble in water, care should be taken not to use too much water. In order to have comparable results, the same quantity of water should be used for all detns (Ratio 1:1)
18) Collect all the wash waters into a large separatory funnel and let them stand overnight in a cool place for further separation
19) Transfer the washed NG, slightly turbid due to the presence of moisture, to a tared dish and place it in a Ca chloride desiccator
20) If the liquid becomes clear, weigh the dish the next day, otherwise continue drying
21) The separation of small amounts of NG left in the acid and wash waters is usually

done the next day
22) Run the acid out of the 500ml funnel and wash the surface of the remaining NG in the funnel with a small amount of water without stirring the mixt
23) Run this NG into a small tared dish and weigh it. Deduct 10% from the weight to allow for the presence of some acid
24) There might be drops of NG stuck to the sides of the separatory funnel contg the wash waters. By using a rubber policeman on a glass rod, these drops can be collected on the bottom and drawn off into a small tared dish, dried over Ca chloride and weighed
25) The combined weights of NG (20, 23 & 24) give the total NG obtd

Calculation:

$$\% \text{ Yield} = \frac{\text{Total Wt of NG} \times 100}{\text{Wt of glycerol taken}}$$

When glycerol is nitrated for use in military smokeless propellants, the main requirement is usually not the yield of NG, but its stability and other properties, as required by the specifications. If this is the case, a smaller sample can be used (10g), but the nitrating acid should be prepd from technical acids used in the plant and not from cp acids

Following is a procedure originated by Hercules Powder Co and adopted at Picatinny Arsenal with slight modifications. All operations should be conducted behind safety glass

Reagents:

Nitroglycerin Mixed Acid. Use a mixed acid from a regular lot of acid used in the plant nitration of glycerol and which meets the current requirements for this acid

Sodium Carbonate Solution. Dissolve 10g of sodium carbonate in 90ml of distd water

Apparatus (See accompanying Figure G18)

A. 1 × 7-inch centrifuge tube immersed to half its height in an ice-water bath held in a 2-liter container E and held in place by a rubber covered clamp

B. 50-ml dropping funnel graduated or marked so as to readily indicate the delivery of 10g glycerol, and having a stem so bent and drawn out as to drop the glycerol directly onto the acid over 20–25 mins

C. Thermometer reading 30–120°F with bulb just under the acid surface

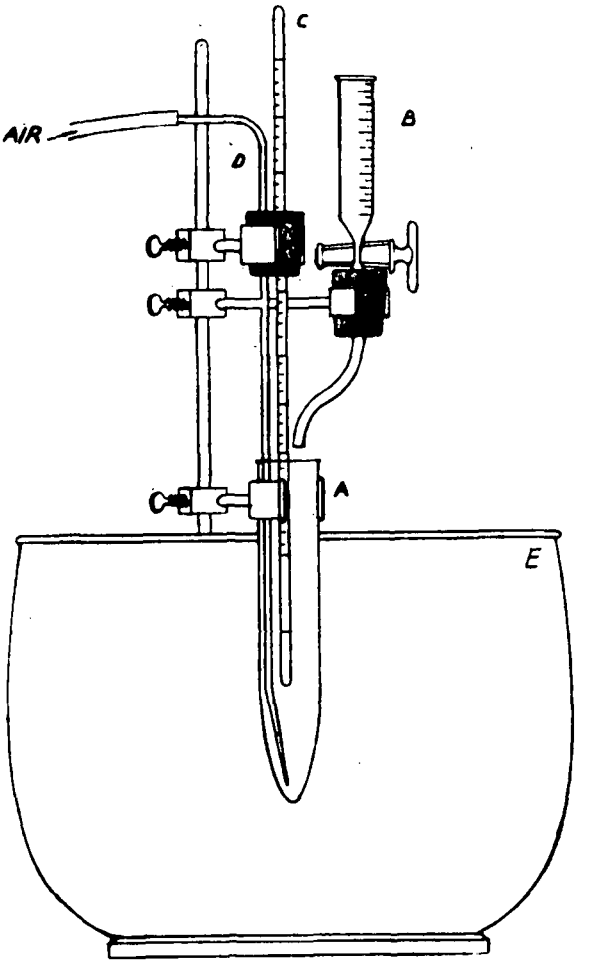


FIGURE G18

D. 5-mm glass tubing drawn down to 1-mm and angled at the end so that the tip is near the bottom of the tube, to serve as an air agitator

E. 2-liter container for ice-water bath (Not shown in Fig G18)

F. 100-ml separatory funnel with short stem, to serve as separator of NG from the spent acid

G. 1000-ml tall form beaker

H. Two 250-ml graduates

I. Two 50-ml conical separatory funnels having short rubber tubing with pinchcock closures instead of glass stopcocks

J. Ring stand

K. Pair of pliers

L. Pair of canvas gloves

Procedure:

Transfer 80g of mixed acid to A and lower into the ice bath so that the acid and bath levels coincide, the thermometer and agitator are positioned as indicated above, and the dropping funnel stem is about 1 inch above the acid. Using vigorous agitation cool the acid to 45–50°F, then start adding the glycerol at 1 drop/10 seconds onto the point of greatest agitation, proceeding thusly until the 10g of glycerol has been added, providing that the temp does not rise above 60°F and that no red fumes appear. Should either of these indications occur, immediately cease the flow of glycerol. This action not proving to have the desired effect, don gloves, break the agitator above the reactor with pliers and then lower the reactor until it may be tipped to drown the contents in the bath E. Then leave the area promptly

Note: Amt of less than 80g mixed acid per 10g glycerol tends to give an explosively unstable spent acid

On completing a normal reaction, raise the funnel out of the way, and transfer the thermometer and agitator to the 1000-ml beaker containing 750ml of cold water

Pour the spent acid–Nitroglycerin (NG) mixture into a 100ml separatory funnel (F) and allow to stand until a good line of separation is obtd. Rinse the nitrating tube, immediately after employing, by filling with cold water, pourin g this rinse water into the beaker contg the thermometer and air tube. Withdraw the spent acid in the 100-ml separatory funnel thru the stopcock into a beaker

Note: This stopcock must be greased after each use and securely held in place by a suitable means

Transfer the NG thru the stopcock, to a 250-ml graduate contg 10ml of water, and place the graduate and contents in a water bath. Insert the thermometer and agitator assembly used in the nitration into the graduate and agitate by passing air thru the charge for 10 minutes maintaining the temp of the charge at 110°F. Pour the charge into a funnel fitted with a piece of rubber tubing and a clamp and allow to settle for 10 minutes. In the meantime decant the water in the liter beaker and transfer any NG present to the charge in the funnel

Note: Do not pour the wash water into a sink!

Insert the thermometer and air agitator assembly into a 250-ml graduate contg 10ml of 10% soda ash (sodium carbonate) soln and agitate the soln. Transfer the NG to the graduate and agitate the mixture for ten mins at 110°F. Pour the charge into the funnel fitted with rubber tubing and after separation takes place, test the soda ash wash water with phenolphthalein paper. If the water is not alkaline, repeat the wash with soda ash soln. Allow the charge to settle for 30 minutes, then filter the NG by means of a funnel fitted with four thicknesses of Whatman No 41 filter paper or equivalent. The NG should now be clear; if not, repeat the filtering operation. Determine the pH value of the NG in accordance with the method described below. If the pH value is not within the prescribed limits, rewash with water or soda ash as required

Collect NG in a rubber-stoppered bottle and save for testing

Tests for NG:

Nitrogen Content

Determine the nitrogen content by duPont-Lunge nitrometer as described in Spec JAN-N-246 and in Vol 1 of Encycl, pp A373 to A376. It is described here as Test 4.3.3: **Determination of Nitrogen in NG,** under **Test Methods and Procedures.** Test 4.3.3.1 is "Standardization of Nitrometer", while Test 4.3.3.3 is the "Procedure with Nitrometer"

Calculation

As the sample of NG was dried only by filtering thru paper, it is assumed that it still retains an average moisture of 0.17%. By introducing a factor 0.9983 in the equation:

$$\% N = \frac{\text{Nitrometer Reading}}{0.9983 \times \text{Wt of sample}}$$

the nitrogen content of the dry NG is arrived at. Minimum for % N is 18.40

Stability of NG by KI Test at 82.2±1° (Abel Test)

Conduct the test in the same manner as called for by the Abel Test with the exception that a temp of 82.2±1° is maintained. The test is described in Specification JAN-N-246B. Minimum requirement is 10 mins as Test 4.3.4, entitled "Determination of Stability of NG", which follows the Test 4.3.3.3, "Procedure with Nitrometer"

Note: Although Spec JAN-N-246 allows a minimum of 10 mins, a minimum of 15 mins is preferred, and, if possible, should be attained. However, if the sample has a KI test at 82.2 ° lower than 15 mins plus but greater than 10 mins plus, the lot may be accepted

Neutrality Test:

The usual method of testing neutrality by means of litmus or phenolphthalein paper is not applicable when the pH of the material is around 7.0. Litmus paper does not turn red until the acidity is between approx pH 4 and 6, and phenolphthalein paper does not change color until the acidity is between approx pH 8 and 10

As freshly washed NG should show a pH of at least 7.0, the most accurate method for this detn is by means of a pH meter. However, if no pH meter is available, a colorimetric method based on the use of La Motte indicators supplied by La Motte Chemical Products Co of Baltimore, Maryland is recommended. This method is rapid and accurate to at least ± 0.05 pH unit

Procedure:

Prepare in advance about 10ml of water with pH exactly 7.0 by neutralizing distd water with NaOH. For testing the water, use a pH meter or the La Motte colorimetric method. Store the water in a Pyrex bottle with a ground glass stopper. If the colorimetric method is used, proceed as follows:

By means of a graduated pipette provided with a rubber suction bulb, transfer 2ml of NG to a La Motte Co comparator tube and add neutral water to the mark on the tube. After this, add 0.5ml of La Motte Co bromthymol indicator, close the comparator tube with a clean cork stopper and shake

Compare the color of the soln with the standards provided with La Motte Block Comparator

The color should be between standards of pH 6.9 and 7.1. However, some plant practices permit a pH of 7.8

Destruction of Waste NG

Any NG left after tests or in waste waters should be mixed, by means of a hard rubber spatula, with sawdust in a waste bucket and sent to the burning grounds. All the apparatus which contained NG should be cleaned with acetone and the washings mixed with the sawdust

If any NG id spilled, it should be cleaned up immediately. To do this, mop up first with a wet cloth which should then be put into the sawdust; then wash with one of the NG removers ("killers" or "destroyers") and finally wash the area with warm water and soap powder

The following NG destroyer is commonly used at Picatinny Arsenal and elsewhere: water 23ml, alcohol (95%) 69ml, acetone 20ml, sodium sulfide 7g. It takes 17ml of this soln to destroy 1 g of NG in 2 mins

Refs for Glycerol Tests and Analyses:

1) Naoúm, NG (1928), 25–40, 229 2) J.W. Lawrie, "Glycerol and Glycols", Reinhold, NY (1928) (ACS Monograph No 44) 3) T. Berth, ChemZtg **51**, 575(1927) & CA **22**, 869(1928) 4) (?) Riesener & (?) Kessen, ChemZtg **52**, 243(1928) & CA **22**, 2057(1928) 5) L.W. Bosart & A.O. Snoddy, "Specific Gravity of Nitroglycerin", IEC **20**, 1377(1928) 6) Hercules Powder Co Manual G50, Revised 4/21/43 7) C.S. Miner & N.N. Dalton, eds, "Glycerol", Reinhold, NY (1953) (ACS Monograph No 117), pp 167–237, 101 refs (by J.B. Segur) 8) K. Namba, KôgyôKayakuKyôkaishi **23**, 76–7 (1963) & CA **63**, 9735(1965)(Calculation of the heat of reaction in the nitration of glycerin)(The values are given in CA) 9) Kirk & Othmer **10**, 627(1965

Glycerol (or Glycerin) Acetates. See under Acetins and Derivatives in Vol 1 of Encycl, pp A31-L to A33-R

Glycerol (or Glycerin) Acetate Dinitrate. See Dinitroacetin in Vol 1, p A33-L and as Glycerol Dinitrate Monoacetate in Blatt, OSRD **2014**(1944)

Glycerol (or Glycerin)-α,α′-bis-[2,4-dinitrophenylether],
$(O_2N)_2C_6H_3.O.CH_2.CH(OH).CH_2.O.C_6H_3(NO_2)_2$; mw 425)09, N 13.18%; yel crysts (benz-eth), mp 79°; sol in alc & hot benz. Prepd by heating the silver salt of 2,4-dinitrophenol

with epichlorohydrin in alc
Refs: 1) Beil **6**, (126) 2) M. Brenans, Bull-SocChFr [4] **13**, 529(1913)

Glycerol (or Glycerin) Chlorohydrin and Its Nitrated Derivatives. See under Chloropropanediol and Derivatives in Vol **3**, pp C265-L to C267-L. The dinitrate is described in Blatt, OSRD **2014**(1944) as Glycerol Monochlorohydrin Dinitrate or α-Dinitrochlorohydrin

Glycerol (or Glycerin) Diacetate and Its Nitrates. See Diacetin and Itd Nitrates in Vol **1**, p A33-L, under Acetins and Derivatives

Glycerol (or Glycerin) Dichlorohydrin and Its Nitrated Derivatives. See under Dichlorohydrin and Derivatives in Vol **5**, pp D1211-L to D1212-R and in Blatt, OSRD **2014**(1944), as Glycerol Dichlorohydrin Nitrate

Glycerol (or Glycerin)-α,γ-diethylether-β-nitrate, $H_5C_2.O.CH_2.CH(ONO_2).CH_2.O.C_2H_5$; mw 193.20, N 7.25%; liquid, bp 169°(dec). Can be prepd by dropwise addition of the diether to mixed acid at 0°

It is an expl, which easily decomposes on heating and does not seem to be suitable as military expl (Ref 3)
Refs: 1) Beil **1**, 515 2) E. Paternò & T. Benelli, Bass **39** (II), 312(1909) & CA **4**, 381 (1910) 3) A.H. Blatt & F.C. Whitmore, OSRD **1085**(1942)

Glycerol (or Glycerin) Dinitrate. See under Glycerol (or Glycerin) Nitrates and in Blatt, OSRD **2014**(1944)

Glycerol-α-[2,4-dinitrophenylether], $(O_2N)_2C_6H_3.O.CH_2.CH(OH).CH_2OH$; mw 258.19, N 10.95%; crysts, mp 85°; sol in w, alc, eth & acet. Prepd from glycerine, 4-chloro-1,3-dinitrobenzene and KOH
Refs: 1) Beil **6**, 255 & [244] 2) C. Willgerodt, Ber **12**, 766(1879)

Glycerol-α-[2,4-dinitrophenylether] Dinitrate (Dinitryl) or **1-(2,4-Dinitrophenoxy)-2,3-propane Dinitrate,** $(O_2NO)CH_2.CH(ONO_2).CH_2.O.C_6H_3(NO_2)_2$; mw 348.19, N 16.1%, OB to CO_2 −50.5%; crysts, mp 124°. This expl compd originally prepd in 1909 in Germany (Ref 2) was also prepd (accdg to G.D. Clift) during WWII by Hercules Powder Co from ortho- or para-nitrophenoxide and glyceryl monochlorohydrin, followed by nitration; it was also prepd by nitration of glyceryl-α-phenyl ether. It proved to be an expl slightly more powerful than PA and of nearly the same sensitivity to impact. Can be initiated by No 6 cap
Refs: 1) Beil, not found 2) Coll, Zentralstelle für wissensch-techn Untersuchungen in Neubabelsberg, Annual Rept (1909) 3) Blatt, OSRD **2014**(1944) 4) G.D. Clift, Private Communication (1960)

Glycerol-α,α′-dipicrylether-β-nitrate, $O_2N.O.CH(CH_2O.C_6H_2.N_3O_6)_2$; mw 559.28, N 17.53%, OB to CO_2 −59.1%; lt-yel solid, mp 158–62°(dec). Prepd by mixed acid nitration of glycerol-α,α-diphenylether. It is expl
Refs: 1) Beil **6**, (141) 2) Dinamite Nobel, SA, GerP 294813(1916)

Glycerol (or Glycerin) Ethers, Nitrates of. E. Paterno & T. Benelli, Gazz **39**, 312(1909) & JSocChemInd **28**, 1168(1909); CA **4**, 381(1910), prepd the nitroderivatives of α-dimethyl and α-diethyl ethers of glycerol by the action of methyl- or ethyl-alcoholic solns of KOH on epichlorhydrin and then nitrating the resulting ethers by nitric-sulfuric acid misture. They obtained:
Dimethyl Glycerol Ether Nitrate, $(H_3C.O)CH_2.CH(ONO_2).CH_2(OCH_3)$; colorless liquid, mp about −15°, bp +180°; sol in common organic solvents; burns readily without expln when ignited by flame
Diethyl Glycerol Ether Nitrate, $(H_5C_2O)CH_2.CH(ONO_2).CH_2(OC_2H_5)$; colorless liquid, mp below −75°, bp 168–70°. Both the dimethyl and diethylnitrate compds readily gelatinize NC and may also serve as antifreeze

addns to Dynamites. When added to NG, they diminish its power without diminishing its sensitivity appreciably

Glycerol Formal, $HO.\underline{CH.CH_2.O.CH_2O.CH_2}$ and $HO.CH_2.\underline{CH.O.CH_2.O.CH_2}$; mw 104.10. Exists in two isomeric forms, both prepd in 1896 (Ref 2) and again in 1930 (Ref 3) by heating formaldehyde and glycerol in presence of HCl. It is a liquid which boils at 195° and proved to be a good solvent for cellulose esters, resins, etc (See also Ref 4)

Refs: 1) Beil **19**, 63 & [69] 2) M. Schulz & B. Tollens, Ann **289**, 29 (1896) 3) A. Fairbourne et al, JSCI **49**, 1069 (1930) 4) J.F. Walker, "Formaldehyde", Reinhold, NY (1953), 141

Glycerol Formate (or Formiate) Dinitrate. See Formylglycerol Dinitrate or Formin Dinitrate, under Formylglycerol or Formin and Derivatives in this Vol, p F173-R

Glycerol-Glucose and Glycerol-Lactose Nitrates. See Glucose-Glycerol and Lactose-Glycerol Nitrates

Glycerol-glycol-ether Trinitrate, $(O_2NO).CH_2.CH(ONO_2).CH_2.O.CH_2.CH_2(ONO_2)$; mw 271.14, N 15.50%, OB to CO_2 −26.6%; liq, sp gr 1.481, n_D 1.4685 at 20°, heat of combustion at const vol 2.4kcal/gm. Prepd by nitration of the parent triol ether at 20° with mixed acid or 99% nitric. Functions as a gelatinizer (presumably for NC, Ref 4). Much less volatile at 80° than NG, lying between EDNA and ethylenenitramine nitrate (Ref 3)

Refs: 1) Beil, not found 2) L. Médard & M. Thomas, MP **37**, 129–38 (1955) & CA **51**, 716 (1957) 3) G. Desseigne, MP **39**, 181–90 (1957) & CA **52**, 21107 (1958) 4) G. Desseigne, FrP 1127647 (1956) & CA **53**, 16970 (1959)

Glycerol-α,α′-hexanitrodiphenyl Ether-β-nitrate; Glycerin-α,α′-dipicrylether-β-nitrate or **Nitroblycerol Dipicrate,** $(O_2N)_3C_6H_2.O.CH_2.CH(ONO_2).CH_2O.C_6H_2(NO_2)_3$; mw 559.30, N 17.57%, OB to CO_2 −50%; lt yel solid, mp 158–62°, bp – dec at 174° c Can be prepd by nitrating glycerol-α,α-diphenylether with mixed acid

It is an expl nearly as powerful as PA, as detd by the Trauzl Block Test and is very insensitive to impact, as detd by Kast Apparatus

Refs: 1) Beil **6**, (141) 2) Coll, Zentralstelle für wissensch-techn Untersuchungen in Neubabelsberg, Annual Report (1909) 3) Dynamit AG, BritP 24352 & CA **24**, 352 (1916) 4) Blatt, OSRD **2014** (1944)

Glycerol (or Glycerin) Lactate Trinitrate. See Glycerol (or Glycerin) Monolactate Trinitrate

Glycerol (or Glycerin) Methylether Dinitrate. See Glycerol (or Glycerin) Monomethylether Dinitrate and in Beil **1**, (272)

Glycerol (or Glycerin) Monoacetate and Its Dinitrate. See Dinitromonoacetin in Vol **1**, p A33-L, under Acetins and Derivatives

Glycerol Monoacyl Derivatives were prepd before WWII by IGFarbenindustrie AG, FrP 848740 (1931) & CA **35**, 5912 (1941), on heating *Glycidol Nitrate*, $\underline{CH_2.O.CH.CH_2(ONO_2)}$ with an org acid in presence of a catalyst. For example, *Glycerol Formate Nitrate*, $OHCHO.C_3H_5(OH)(ONO_2)$, bp 125–26°, was obtd by heating Glycidol Nitrate (238 parts) with formic acid (100p) in presence of about 1% catalyst, duch as $FeCl_3$. The resulting product was suitable as an antifreeze addn to NG Dynamites

Glycerol-α-monomine. See 1-Amino-2,3-propanediol and its nitrated derivatives in Vol **1**, p A251-L & R

Glycerol-β-monoamine. See 2-Amino-1,3-propanediol and its nitrated derivatives in Vol **1**, p A251-L & R

Glycerol Monochlorohydrin Dinitrate. See in Vol 3 of Encycl, pp C265-R & C266, under Chloropropanediol and Derivatives and in Blatt, OSRD **2014**(1944)

Glycerol Mono-α-[2,4-dinitrophenyl]-ether. See Glycerol-α-(2,4-dinitrophenylether) in this Vol

Glycerol Monoglycolate Trinitrate, $(O_2NO).CH_2.CH(ONO_2).CH_2.O.(COCH_2.ONO_2)$; mw 285.13, N 14.74%, OB to CO_2 −14.0%; oily liq with a viscosity greater than NG. It was prepd by the DuPont Co by nitrating glycerol monoglycolate. It is an expl less sensitive to impact than NG and possessing satisfactory thermal stability; less volatile than NG
Refs: 1) Beil, not found 2) Blatt, OSRD **2014**(1944)

Glycerol (or Glycerin) Monolactate Trinitrate or **Glycerol Lactate Trinitrate (GLTN),** $(O_2NO).CH_2.CH(ONO_2).CH_2O.C(O).\underset{\displaystyle CH_3}{\underset{|}{CH}}(ONO_2)$; mw 299.16, N 14.04%, OB to CO_2 −29.4%; oily liquid, slightly more viscous and less volatile than NG; sp gr 1.47, n_D 1.464 at 25°; nearly insol in w; miscible with eth, 2/1–eth/alc & acetone

Was first prepd in 1931 by nitrating glycerol lactate with mixed acid (Ref 2). The prepn of glycerol monolactate by heating glycerol with equimolecular proportion of a lactic acid ester of an alcohol boiling below 100°(such as ethyl lactate) was patented in 1936 (Ref 3). Blatt (Ref 4) described heating of glycerol with lactic acid for 30 hrs at 110°, followed by nitration. Accdg to AMCP 706–177 (Ref 7), glycerol monolactate was prepd by heating a glycerol-lactic acid mixt (contg 4% excess lactic acid) at 116° for 112 hours with dry air bubbling thru the liquid. The product which contd 0.67% free acid was carefully mixed with six parts of 40/60–HNO_3/H_2SO_4 maintained at 20°, stirred for 1 hr cooled to 5°, and poured on ice. It was extracted with ether, water-washed, adjusted to pH 7 by shaking with a Na bicarbonate soln, and again water-washed three times. It was then dried with Ca chloride, filtered and freed of ether by bubbling with air until minimal loss of wt was obtd. The product had a nitrate-nitrogen content of 13.43% (theory 13.04%). Another batch, prepd from glycerol lactate contg 6.5% excess glycerol had a nitrate-nitrogen content of 14.30%, corresponding to a mixt contg 5.5% NG. It is not considered practicable to prepare pure GLTN

The following are properties of GLTN:
Brisance by Sand Test. 13.1g sand crushed by 0.4g sample (vs 48.0g for TNT)
Explosion Temperature. 223° in 5 secs (vs 475° for TNT)
Friction Pendulum Test. Unaffected by fiber or steel shoe (same as for TNT)
Heat of Combustion. 2407cal/g (vs 3620cal/g for TNT)
Heat Test at 100°. 2.5% loss in 48 hrs, 1.8% in 2nd 48 hrs and no expln in 100 hrs (vs 0.2, 0.2 and no expln in 100 hrs for TNT)
Hydrolysis. 0.021% acid after 10 days at 22° or 0.014% after 5 days at 60°
Impact Sensitivity. BurMinesApp, 2kg wt, 42cm (vs 95–100 for TNT)
Impact Sensitivity. PicArsn App, 1 lb wt – 15 inches for 20mg sample (same as for TNT)
Power by Ballistic Mortar. 114% of TNT (Ref 6)
Vacuum Stability Test at 100°. 5.9cc gas evolved from 5g sample in 40 hrs (vs 0.10cc from TNT)

Macy & Saffitz examined at PicArsn (Ref 5) GLTN as possible expl plasticizer for NC and found it acceptable from most standpoints, but a less effective gelatinizer than NG
Refs: 1) Beil, not found 2) C.M. Stine & C.E. Burke, USP 1792515(1931) & CA **25**, 2294(1931) 3) R.H. Locke, BritP 456525 (1936) & USP 2087980(1936) 4) Blatt, OSRD **2014** (1944) 4a) ERL, Bruceton, Pa, OSRD **5746**(1945) 5) P.F. Macy & A.A. Saffitz, "Explosive Plasticizers for Nitrocellulose", PATR **1616**(1946) 6) ADL, PureExplCompds, Part 1 (1947), p 149 7) W.R. Tomlinson, Jr & O.E. Sheffield, "Properties of Explosives of Military Interest", AMCP **706–177**(1967) (formerly PATR **1740**), pp 140–42

Glycerol (or Glycerin)-α-monomethylether Dinitrate,

$H_3C.O.CH_2.CH(ONO_2).CH_2(ONO_2)$; mw 196.12, N 14.29%, OB to CO_2 – 40.8%; solid, sp gr 1.374 at 15/15°, mp 24°, bp 124° at 18mm; sol in methanol, alc, eth and benzl insol in CS_2 & petr eth (Ref 2). Was prepd by nitrating glycerol-α-monomethylether with mixed acid contg 38.6% nitric acid and 59% sulfuric acid, during 20–25 mins

Its properties: Impact Sensitivity by FI >120% PA, which means very insensitive. Mixt of 3 parts the dinitrate and 1p kieselguhr detonated by 1kg wt falling from a height >85cm, vs >10cm for a similar mixt of NG and kieselguhr. Power by Ballistic Mortar 76% NG. Power by Trauzl Test 77% NG, 135% PA or 147% TNT. Expls at 182° when heated at the rate of 5°/min

Refs: 1) Beil **1**, (272) 2) D.T. Jones, JCS **115**, 76(1919) & CA **13**, 1149(1919) 3) Blatt, OSRD **2014**(1944)

Glycerol Monostearate (Monostearin), Alpha, $C_{17}H_{35}CO_2.CH_2.CHOH.CH_2OH$; mw 358.55; colorless ndls (from Me alc), sp gr 0.984 at 20/4°, mp 58–59° (capillary tube); insol in w; sol in eth & hot alc; also in oils and hydrocarbons. Can be prepd from stearic acid and glycerol. Can be used as a protective coating for hygroscopic expls & proplnts; produces better candles than paraffin and just as good as wax candles

Refs: 1) Beil **2**, 380 2) F. Hundeshagen, JPrCh [2]**28**, 225(1883) 3) CondChemDict (1961), 541-R; (1971), 421-L

Note: There is no reference in the open literature to a mono- or dinitrated derivative of glycerol monostearate

GLYCEROL (OR GLYCERIN) NITRATES AND DERIVATIVES

1-Nitratoglycerol, α-Glycerol Nitrate, $HO.CH_2.CHOH.CH_2.ONO_2$; mw 137.09, N 10.22%, OB to CO_2 –52.5%; plates (from w), mp 54°, bp 155–60°; sol in w & alc, sl sol in eth. Prepd along with the β isomer by nitrating glycerol. Prior to the publication of Ref 4, it was thought to be the 2-, or β-, nitrate. It is not expl

2-Nitratoglycerol, β-Glycerol Nitrate, $HO.CH_2.CH(ONO_2).CH_2OH$; mw 137.09, N 10.22%, OB to CO_2 –52.5%; colorless prisms (from w, alc or eth), sp gr 1.40, mp 58–9°, bp 155–60°; sol in w & alc; dif sol in eth. Prepd by treating 2,3-epoxy-1-propanol with dil nitric acid. Prior to the publication of Ref 4 it was thought to be the 1-, or α-, nitrate. It is not expl

Refs for Glycerol Mononitrates:

1) Beil **1**, 514, 515, (272) & [591] 2) Hanriot, AnnCh [5] **17**, 118(1879) (β) 3) W. Will, Ber **41**, 1120(1908)(α, β) 4) E.Q. Adams, JACS **42**, 1321(1920) 5) Naoúm, NG(1928), 161–67 6) H.F. Bluhm, USP 3256214(1966) & CA **65**, 9132(196))(Reaction with polyisocyanates to form polyurethanes) 7) J.M. Trowell, AnalChem **42**(12), 1440–2(1970) & CA **73**, 111480(1970) (Gas chromatographic analysis of partial break down of NG in double-base proplnts)

1,2-Dinitratoglycerol, α,β-Glycerol Dinitrate, $HO.CH(ONO_2)CH_2.ONO_2$; mw 182.09, N 15.38%, OB to CO_2 –17.6%; oil, bp 146–8° at 15mm Hg, does not solidify on cooling; sol in w, alc, eth. Prepd by mixed acid nitration of glycerol below 20°

1,3-Dinitratoglycerol or α,α'-Glyceroldinitrate, $O_2N.O.CH_2.CHOH.CH_2.ONO_2$; mw 182.09, N 15.38%, OB to CO_2 –17.6%; crysts, mp 26° (with 1/3 H_2O); sol in w, alc & eth; in the anhydrous state it is an oil, fr p –40°, sp gr 1.47 at 15°, bp 146–8° at 15mm Hg (slight dec); detonates on heating. Can be prepd by mixed acid nitration of glycerol below 20°

Commercial Glycerol Dinitrate, liq, sp gr 1.517 at 20°, bp 146° at 15mm (slight dec); sol in w, alc, eth, chlf & NG; hygroscopic. Prepd by incomplete nitration of glycerol using straight nitric acid (sp gr 1.5) in a ratio of 3–4p to 1p glycerol, giving an isomeric mixture (Ref 5, p 164, by A. Mikolajczak). About 20% lower yield results by nitrating with 5p of mixed acid contg nitric acid 23% & sulfuric acid 68% at or below 20° (Ref 4). It is an expl 81–83% as powerful as NG and about 70% as brisant by the lead block compression method. It is slightly less sensitive to impact than NG and slightly more stable to heat

NC gelatinized with it is sticky, hygroscopic, and less elastic compared with NG gelling

The DNG explodes according to the equation:

$C_3H_5(OH)(ONO_2)_2 \rightarrow 2CO_2 + CO + 2H_2O + N_2 + H_2$ developing a heat of expln of 1304.4cal/g at C_v and H_2O (l), or 1201cal/g with H_2O (v) (Ref 5, p 169). The heat of formation is 116.7kg cal/mol

When heated slowly DNG can be evaporated without expln or deflagration, but when heated rapidly in the open it deflagrates with a slight noise. For this reason expls containing DNG and no NG are much less sensitive to heat

Uses: DNG has been used as an antifreeze in NG Dynamites. Advantage was taken of its lower mechanical sensitivity and much lower thermal sensitivity compared with NG in the prepn of these dynamites manufd in France (Ref 3):

1) DNG 60, AN 35 & NC 5%
2) DNG 40, KNO_3 51, curcuma (meal) 4.4, cellulose 4 & NC 0.6%
3) DNG 35, AN 38.4, Na oxalate 12, NH_4Cl 9.4, TNT 2.5, cellylose 2 & NC 0.7%
4) DNG 58, KNO_3 36, cellulose 4 & NC 2%

Refs on Glycerol Dinitrates:

1) Beil **1**, 515, (272) & [591] 2) W. Will, Ber **41**, 1110(1908) 3) M. Dautriche, MP **14**, 185(1906–07) & CA **3**, 2053(1909) 4) Marshall, **1**(1917), 238 & **2**(1917), 704, 706 5) Naoúm, NG(1928), 161–70 6) G. Desseigne, MP **45**, 103–114(1963) & CA **63**, 5516(1965)(Prepn of dinitrates and derivs of glycerol) 7) I. Dunstan et al, JCS **1965**(Feb), 1319–24 & CA **62**, 8992 (1965)(Prepn of dinitrates and derivs) 8) V. Styblova, CeskNeurol **29**(12), 1440–2(1970) & CA **73**, 111480(1970)(Gas chromatographic analysis of double-base proplnts for partial breakdown products of NG)

Glycerol Acid Dinitrate, $O_2N.O.CH_2.CH(ONO_2)C(O)OH$; mw 196.09, N 14.28%, OB to CO_2 0.0%; wh crysts (from benz or eth/ligr), dec 117°; sol in w, alc & eth. Prepd by treating 2,3-dihydroxypropanoic acid with mixed acid at −12°

Refs: 1) Beil **3**, 397 2) H. Duval, CR **137**, 573(1903)

Glycerol Methylether Dinitrate. See Glycerol (or Glycerin) α-monomethylether Dinitrate

GLYCEROL TRINITRATE OR NITROGLYCERIN (NG) (Nitroglycérine in Fr, Nitroglyzerin in Ger, Nitroglicerina in Ital, Nitoroguriserin in Japan, Nitroglicerina in Span, Nitroglitserin in Russ). See Vol **5**, p D1593-L to D1594-L for many of the physical props normally found following a compound name

Additional Physical Properties. NG is, when pure, a clear, colorless, oily liquid having no odor and a sweet burning taste. Two cryst forms are known: a glassy, triclinic **labile** one with fr p of 2.1°, mp of 2.8°, heat of crystn of 5.2cal/g, heat of conversion to the **stabile** form of 28.0cal/g; a dipyramidal rhombic **stabile** one with fr p of 13.2°, mp of 13.5°, heat of crystn of 33.2cal/g. The tendency to supercool results in a narrow range of reported fr p and mp values. NG has an apparent bp of 145°, which is merely the temp at which non-expl decompn becomes vigorous enough to resemble boiling. Apparently some decompn occurs even during high vacuum distillation, but some reported values are 125° (2mm Hg) and 180°(50mm Hg), NG is slightly volatile with steam, requiring one liter of steam distillate to carry over 5ml of NG. The vapor pressure of NG is (°C, mm Hg): 15, 0.00130; 25, 0.00177; 35, 0.00459; 45, 0.01294; 55, 0.03587 (decompn begins at 50–60°). Relative viscosity of NG (time in sec to run out a 10-ml pipet at 20°) is 15, compared with 6 for water and 540 for glycerol. The solubilities of NG are: 1.8g per liter of w (20°) & 2.5g per liter (50°); 37.5g per 100g abs alc (0°) & 54g per 100g abs alc (20°); 40g per 100g 96% alc (20°); 1.25g per 100ml CS_2 (RT) – this low sol in CS_2 is taken advantage of in extracting other expl ingredients from NG mixts; 2ml per 100ml CCl_4; 20p per 100p trichloroethylene; 12p per 100p Gc (20°) & 20p per 100p Gc (80°); Solubilities of interest in NG are: 35g DNT per 100g NG (20°); 30g TNT per 100g NG (20°). NG and NGc are misc in all proportions and form two eutectics: a) 29% stabile NG/71% NGc melting at −29°; b) 39% labile NG/61% NGc melting at −33°(Ref 14b). Lewis (Ref 14a) used published data on the temp dependence of density, surface tension and vapor pressure to calculate values for the critical constants of NG: T_c 720°K, P_c 35.2 atm. He gives for the surface tension at any temp T(°K): γ(dynes/cm) = 94.58[(720−T)/720

Chemical Properties. NG reacts chemically as a nitrate ester, saponifiable to glycerol by alkalies (preferably with peroxide present), and hydrolyzed by nitric acid to glycerol. In general, side products tend to be produced resulting from cleavage of the C–C bonds and oxidation of the alcohol functions

Explosive Properties of NG. See also Vol **5**, p D1593-R and D1594-L. In general, bulk NG is difficult to ignite by *flame* or *heat*, the ease of ignition improving as the NG layer becomes thinner; NG may expl instead of igniting if large quantities are subjected to localized, sudden heating. When frozen, NG is rather insensitive to *shock*, and in the liquid form becomes increasingly sensitive as its temp increases. However, NG is most shock sensitive when crysts are in contact with liq. Other expl props are:

Brisance by Hess Pb Block Test (Ref 8a). The stabile cryst form was shown to have much greater brisance, and to have higher sensitivity than the labile cryst or liq forms.

Critical Diameter. A range from 3.9mm at –20° to 1.1mm at 70° is reported by Belyaev & Kurbangalina (Ref 15a)

Detonation Velocity. In 22mm ID glass tubes (26mm OD) with 7000m/s PA fuse, deton vels of 9150m/s, 0m/s, 1165m/s were obtd for the stabile, labile, and liq forms, resp. Use of a 20g Tetryl booster gave 9100m/s for the labile form, while use of 10–15g of the stabile form as a booster gave 8750m/s for the liq form

Electrical Sensitivity. At 50–60° NG liq or satd in filter paper does not ignite or expl from a 13kV spark from an 8 μF condenser (Ref 8b)

Gap Sensitivity. Blends of NG 15 and inert salt 85% (the latter sifted first thru 20 mesh/sq cm screen) were packed in two identical 30–32mm diam paper tubes, and placed end-to-end on dry sand a definite distance apart. Detonation of one tube resulted in detonation of the other at maximum gaps (in cm) for these salts of: NH_4Cl 25, NaCl 11, $NaHCO_3$ 10. Diammonium sulfate or carbonate, chalk or talc did not transmit the detonation even at zero gap. The particle size of the salts was of some importance, eg, for NaCl the optimum size was 0.10–0.12mm in greatest dimension (Ref 10b)

Impact Sensitivity. Values determined by the drop weight method have the usual dependence on the instrument used, also having great dependence on the area of the impacted NG, the smoothness of the two surfaces involved, and the aeration of the NG. A force of at least 1000g cm was found necessary when using a 5-cm diam weight, but a greater figure was found with a 2.5-cm diam. Even the slightest dents in the anvil will greatly increase the sensitivity of this test. Detonation is attributed to thermal ignition from compressed gas bubbles (the degree of compression being higher in a dent than in the body of the NG) (Refs 10a & 11a)

Power by Trauzl Test. Cavities of 390, 560 and 518cc were obtd for the stabile, labile and liq forms, resp (Ref 8a)

Temperature of Detonation. The value 4850°K is given for the conditions of NG=3CO +2.5 NG = $3CO_2$+2.5H_2O(g)+1.5N_2+0.5 O_2 and a resultant pressure of 10000 atm, or 1100°K at 1 atm (Ref 16)

Historical. NG was first prepd by Ascanio Sobrero in 1846 in Italy by adding glycerol to MA (mixed acid, usually nitric-sulfuric) at 10°. An expln involving a very small amount discouraged him from further investigation. NG prepd by him still exists, stored under w, and washed occasionally. Sobrero referred to the material as "piroglycerina". NG was, as a dil alc soln, used medicinally, going under the name "glonoin" (See also in Vol **5**, p D1585-R, and D1584-L to D1586-R for pre- and non-Nobel expl uses for NG). In 1863 Alfred Nobel, along with his father and brothers set up a laboratory-plant to make and study NG. Their initial methods involved "cold" and "warm" techniques. In the former, portions of glycerol were added to cooled MA in stone jugs with hand stirring and ice-w cooling, followed by drowning in cold w. The warm method was to simultaneously add streams of glycerol and cooled MA to a conical Pb vessel having perforations in the constriction which allowed the NG and spent acid to flow continuoudly into the ice-cooled drowning bath beneath. The capacity of the latter determined the size of the batch. (Certain "continuous" methods developed in the 1960's are based on this same idea.) Temp in the cone was maintained at 45–65°C by adjustment of the two streams. In either method, the NG was then drained from the tank, washed with warm w, then with warm carbonate soln until acid free. The cold method was given preference because of higher yield

Once Nobel began licensing the construction of plants to make NG, they were generally built very close to the site of intended use as transportation of liq NG tended to generate loss of life and property; the shipping of frozen NG as practiced by Mowbray in the USA caused similar problems when the NG thawed prematurely or was not thawed properly. In what is called the *Kopp Process* equipment consisted of a stoneware jug for cooling and washing, a cast iron vessel for mixing, a Pb or porcelain measuring vessel for the glycerol, an iron stirring rod, and a glass funnel with attached rubber hose and stopcock for separating the oily NG from the wash w and dropping into glass flasks. The standard charge was 350g of glycerol slowly added to 2.8kg of stirred MA in an iron vessel kept in the icewater filled jug. The mix was then dumped into the icewater. The aq layer was decanted, the oil washed a few times, and the aq layer decanted again. The wet oil was moved to the funnel for final separation from the w. Total time was 15–20 mins, yield 80–90% of current best. This method, adapted somewhat for "assembly line" procedure, continued into the 1870's, even in large plants. In 1868 Mowbray in Massachusetts introduced compressed air agitation .This later lost favor in the USA to mechanical agitation, but was universally adopted elsewhere for decades

The next development was the *Boutmy-Faucher Process* used in Fr until 1882 and for a long time in Eng. The novelty was to dispense with the need for extensive cooling by preblending the glycerol and sulfuric acid (most of the exothermicity in the making of NG comes from an initial formation of glycerol sulfate). The predissolution occurred in a w-cooled iron trough, with stirring to keep the temp below 40°. The soln was then poured into crocks to cool overnight. Then with cooling and air stirring the MA was added, and the mixture allowed to sit overnight because of very slow separation of the NG. The contents were then drained via a cock. Although yield was higher than in the Kopp Process when everything went right, it was often (enough to cause discontinuation of use) zero because the lengthy contact of spent acid and NG tended to cause explns. The quantities involved were nitric 280 & sulfuric 280 added to glycerol 100 & sulfuric 320 parts

European equipment in the 1920's was still closed Pb cylinders with internal Pb cooling coils and compressed air stirrer, the mixture exiting from a large valve at the bottom. Operating temp was 20–30°, or 12° if –10° brine was used for a slightly higher yield. The contemporary American equipment was all iron and steel cooled by –20° brine to give 2–3° mix temp with mechanical stirring

No attempts to recover the spent acid were made until 1877. Artificial refrigeration in the form of 12° brine was developed over the period 1895–1905. The period 1904–1912 saw the introduction of materials to speed the NG separation time. Naoúm in 1904 was the first entrant in this contribution to the field, patenting the use of liquid paraffins near the end of the glycerol addition, on the basis of 100cc for 250kg glycerol and 1600kg MA. In 1905 C. Reese patented the use of NaF; this turned out to require the presence of silicates, the desired effect being due to the evolution of SiF_4 gas. In praetice NaF/guhr blend was added just before the reaction was over. The paraffin-NaF methods could be combined. Large scale recovery of the nitric acid involved flow of the spent acid down a packed column while superheated steam was rising. The carried-over nitric and nitrous acids were then cooled, or the nitrous first reoxidized to nitric via an air stream

Laboratory Preparation. See Titration Test, under GLYCEROL

Batch Manufacture. The raw materials are mixed acids (MA), mixtures of sulfuric and nitric acids, and "dynamite glycerol", the highest grade of commercial glycerol. The latter must be checked (*first use of each n ew batch*) *for water content* by the Karl Fischer Reagent method and for sp gr by the pycnometer method (1.263 at 15° is the required reading for 100% purity). The MA compn should be 52–55% sulfuric and 47–49% nitric. It should be prepd well in advance of use to allow metallic sulfates to settle out (which otherwise might interfere with NG separation). Lower oxides of nitrogen should be minimal. An important factor in the compn of the MA is the Dehydrating Value of the Sulfuric Acid, known as DVS. This is the ratio of the sulfuric acid to water at the end of the reaction; the water amount will consist of that formed during the reaction and that present in

the raw materials. In practice the DVS is kept between 4.35–4.5 to assure good dehydrating ability thruout the reaction, which maintains the nitric acid at a concn sufficient to insure completion of the reaction (See Vol 3, p D44)

Another factor to consider is the excess nitric required to drive the reversible reaction to completion. Theoretically 1 lb of glycerol needs 2.054 lbs of nitric, but in practice 2.3 lbs is used. The following are representative formulas used in the calculation of the MA compn (based on glycerol containing 1% w):

$$A = \frac{N(0.59/R) + W + L(1.065/D)}{C[(0.01225/D) + 0.01225]} - 1$$

where: A = lbs of fuming sulfuric to be added to 100 lbs of premix
N = % actual nitric in premix
R = ratio of nitric to glycerol
W = % actual w in premix
L = % total NO_2
D = DVS
C = % SO_3 in A

and

$$A = \frac{N(0.59/R) + W - S/D}{C[(0.01225/D) + 0.01225]} - 1$$

where S = % actual sulfuric in premix, and the other letters are the same as defined for the first formula

Refer to Figure G18a (from Ref 14c) for a flow chart of a typical acid prepn plant involving on site manuf of nitric, purchase of 65% oleum sulfuric, and recycling of spent acid; the figures are for MA for NG prepn and would be different for NC, TNT, etc. The pre- or semi-mix consists of a blend of 97% nitric and 40% oleum sulfuric (contg 6% nitric as anti-freeze). Analysis of the pre-mix gives the values for the variables in the two formulas. Then A lbs of the 40% oleum is added to give the final MA blend

Refer to Figure G19 (Ref 14) for a flow chart of a typical batch prepn of NG (or EGDN or EGDN–NG). In the USA the reactor is of steel, equipped with steel cooling coils and mechanical agitation. A 6800 lb charge of MA is run into the reactor with –20° $CaCl_2$–brine-cooled coils and stirring. The glycerol is added in a small stream while closely watching the temp for any tendency to rise. The temp is maintained at 2–3° by cutting off the glycerol as necessary; should this action fail to bring about the desired cooling immediately, the charge is drowned in the water tank beneath the reactor. With adequate stirring the NG does not freeze to the coils as formed because its fr p is lowered by the incorporation of the acids. Stirring is continued for a few mins after the 50–60 mins required to add the glycerol. Then the NG is allowed to separate completely. The lower layer of spent acid is drained off to be recycled or otherwise disposed of, and the NG is run into the neutralizer. An initial 40° w wash removes most of the acid. Then 2–3% sodium carbonate soln neutralizes the residual acid. Finally a concd soln of NaCl breaks any NG–w emulsion. The (tested) neutral NG is then transferred to storage or mix house (Dynamite, Smokeless Powder, etc)

Continuous Manufacture. See Continuous Methodd for Manufacturing Explosives, Vol 3, pp C501-L to C504-R

Desensitization of NG. See Desensitizers and Desensitized Explosives, Vol 3, pp D88-R to D90-L

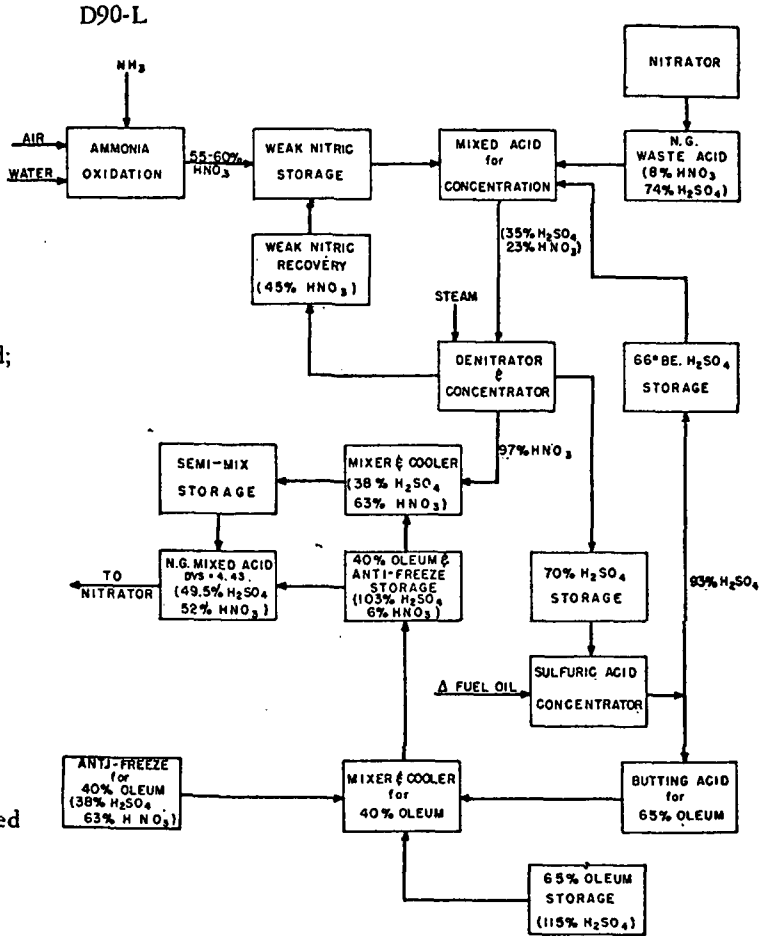


FIG G18a Acid-operation flow chart

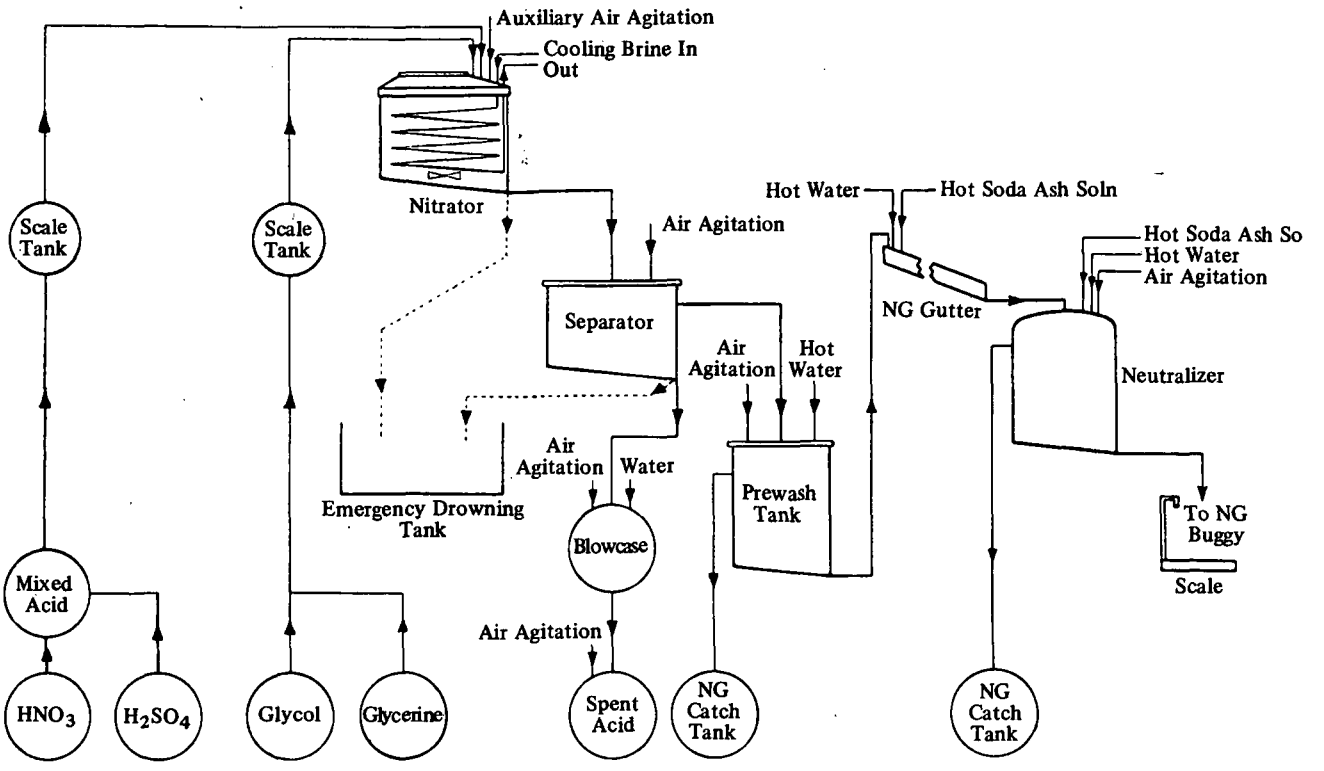


FIG G19 Flow diagram of batch nitroglycerine process

Toxicity of NG. According to Sax (Ref 25), NG is classed as highly toxic (meaning death or permanent injury after very short exposure to a small quantity) after ingestion, inhalation, or skin absorption. Poisoning symptoms consist of headaches and reduced blood pressure, excitement, vertigo, fainting, respiratory rales, and cyanosis. Taken internally it causes death from respiratory paralysis. Under normal manuf conditions no symptoms are observed during the work week (after an initial reaction in most new workers in the form of severe headaches). Over the weekend or vacation periods the worker may have to take small doses of NG to prevent onset of the headaches. The threshold limit value (TLV) as determined by the American Conference of Governmental and Industrial Hygienists (ACGIH) is 0.2ppm or 2.0mg/cu m in air and is good for the atmospheres to be found in NG plants where a mixture of 60–80 EGDN and 40–20 NG is currently being manufd instead of straight NG. In the ACGIH publication (Ref 26) the same TLV is given, but it is also recommended that the value be only 0.02ppm when the exposure is intermittent rather than continuous, as there seems to be some evidence that the deacclimatization process is physiologically dangerous. Good ventilation is a must as the vapor pressure of NG at 20–25° corresponds to about 2ppm

Uses of NG. Aside from the expl uses discussed under Dynamite and Smokeless Powder (NG gelled with NC), the only extensive use for NG is as a decreaser of blood pressure in the treatment of angina pectoris where it is in competition with other polyol nitrates. There is voluminous literature on the pros, cons, and wherefores of this subject

General Refs on NG: 1) Beil **1**, 516, (272), [591] & {2328} 2) A. Sobrero & M. Pelouze, L'Institut **15**, 247(1847) & Ann **64**, 398(1848) 3) O. Guttmann, "Die Industrie der Explosivstoffe", Whittaker & Co, London (1895) 4) R. Escales, "Nitroglyzerin und Dynamit", von Veit, Leipzig (1908) 5) E.M. Symmes, ChemMetEng **25**, 831(1921) & ChemZtr **1**, 3485(1924)(Manuf)

6) A. Schmid, SS **22**, 170, 201 & ChemZtr **II**, 2484(1927)(Manuf) 7) J.W. Lawrie, "Glycerol and the Glycols", ChemCatCo, NY(1928), p 337(ACS Monograph Series No 44) 8) Naoúm, NG (1928) 8a) A.A. Dsershkovich & K.K. Andreev, SS **25**, 353–6, 400–3(1930) & CA **25**, 1674(1931)(The properties of NG isomers)(A review) 8b) H.E. Nash, ArmyOrdn **11**(65), 384–5(1931) & CA **25**, 2294(1931)(Effect of static electricity on Nitroglycerol and related compounds) 9) A. Stettbacher, "Die Schiess- und Sprengstoffe", 2nd ed, Barth, Leipzig (1933) 10) V. Grignard, Ed, "Traité de Chimie Organique", Masson, Paris), Vol **6**(1940), pp 643–652 (P. Pascal, "Industrie de la nitroglycérine") 10a) F.P. Bowden et al, CouncilSciIndResBull No **167**, 44pp(1943) & CA **41**, 3297(1947) 10b) A.I. Gol'dbinder, DokladyAkadNauk **50**, 199–200(1945) & CA **43**, 1567(1949) 11) J. Weir, Nature **158**, 83–5(1946)(Nitroglycerine and Guncotton: A double centenary) 11a) F.P. Bowden et al, ProcRoySoc(London) **A188**, 291–329(1947) & CA **41**, 3626–27(1947) 12) I.H. Favalli, RevBrasilQuim(Sao Paulo) **27**, 79–84 (1949) & CA **43**, 4855(1949) (The history of NG, properties and modern methods of manufacture are described) 13) C.S. Miner & N.N. Dalton, Eds, "Glycerol", Reinhold, NY (1953), pp 362–4, 437–8 (ACS Monograph No 115) 13a) T. Sakurai & Y. Sato, JIndExplsJapan **14**, 111–16(1953) & CA **49**, 11283(1955) (Gelatinization of NC by NG) 14) H.J. Klassen & J.M. Humphrys, ChemEngrgProg **49**, 642(1953) (Diagram of NG batch process) 14a) D.T. Lewis, JApplChem **3**, 154–60(1953) & CA **47**, 7217(1953) 14b) L. Avogadro di Cerrione, Chim&Ind(Milan) **35**, 491–6(1953)& CA **48**, 3029(1954) 14c) G.M. Masson et al, "Detonation and Explosive Phenomena", ProgrRept No **12**, p4, Apr-June, 1956, USBurMines, Pittsburgh, Pa (Temps of deton of NG at low velocites 1200–2000m/sec were betw 3745 & 4237°K. They were generally 600 to 700°K lower than the temps measured at its high velocities of deton) 14d) P.H. Groggins, Ed, "Unit Processes in Organic Synthesis", McGraw-Hill, NY (1958), p 102 15) S. Nauckhoff & O. Bergström, "Nitroglycerin och Dynamit, Bidrag till Fabrikationens Historia", Nitroglycerin AB, Gyttorp, Sweden (1959) & Explosivst **6**, 137 (1960)(Review) 15a) A.F. Belyaev & R.Kh. Kurbangalina, ZhurFizKhim **34**, 603–10(1960) & CA **55**, 15931(1961)(The effect of initial temperature on the magnitude of the critical diameter of Nitroglycerin and Trotyl) 16) A. Schmidt, Explosivst **10**, 115–21 & 133–5 (1962) & CA **58**, 7779(1963) 16a) A.M. Pennie, Explosivst **10**, 213–19, 242–47 & 261–66(1962); **11**, 21–24(1963) & CA **58**, 8843(1963)("Nitroglycerine – A Century of Manufacture", 74 refs) 17) R. Giuliano & M. Rananelli, "Laborazioni che espongono all' azione dei glicoli, nitroglicerina e derivati", Edizioni INAIL, Milan (1962), 92pp & CA **58**, 14622(1963)(Manufacturing which exposes one to the action of glycols, Nitroglycerin and derivatives) 18) F.A. Patty, Ed, IndHyg&Tox, J. Wiley-Interscience, NY, Vol **2**(1963), pp 2085–9 & 2095–6 19) P. Carmichael & J. Lieben, 14th InternCongrOccup-Health, Madrid, Spain **2**, 514–16(1963)(Pub 1964) & CA **64**, 4156(1966)(Sudden death in explosive manufacturing workers – exposed to NG and NGc)(Gives old and new data on chronic toxic effects) 20) H.E. Stokinger et al, JOccupMed **5**(10), 491–8(1963) & CA **60**, 6120 (1964)(Threshold limit values for 1963, including NG) 21 Kirk & Othmer, Vol **8**(1965), pp 602–4, 632–4 22) J.C. Munch & B. Friedland, Ind-MedSurg **34**(2), 143–6(1965) & CA **63**, 1131 (1965)(Glyceryl Trinitrate.I. Acute toxicity) (A review) 23) J.C. Munch et al, IndMedSurg **34**(12), 940–3(1965) & CA **64**, 11749(1966) (Glyceryl Trinitrate.II. Chronic toxicity) (Review with 35 refs) 24) V.P. Zhmurkin, Zh-NevropatolPsikhiat **67**(9), 1336–41(1967) & CA **67**, 115726(1967) (Effect of NG on cerebral vascular tone and pathogenesis of headaches caused by NG) (Conclusion is that headaches from NG are due to swelling of brain veins, lowering of tone in the same, or lack of tone in the same in comparison with increased tone of brain arteries and blood flow therein) 25) Sax, 3rd Ed (1968), pp 969–70 26) ACGIH, "Documentation of the Threshold Limit Values", 3rd Ed (1971), American Conference of Govemmental & Industrial Hygienists (ACGIH), Cincinnati, Ohio, pp 110–111

Description of properties of NG for use in Dynamites is given in Vol **5**, p D1593-L & R

Glycerol Trinitrate, Analytical Procedures. NG, as well as many other noninitiating expls can be identified by making a series of tests, such as described in StdMethodsChemAnalysis (Ref 12a, p 1347), and comparing the results

with those given in Table 32-2 on p 1348. The tests are as indicated below:

Test 1. Place 0.05g of unknown expl in a 5-ml beaker, add 2–3ml of distd w, and stir for 5 min. Observe the color of the liquid. (No color is given by NG, EDNA, NGc, NGu, NC, AN, Ednatol, Amatol, Ammonal and BkPdr). Add a drop of Nessler's reagent and note the color of any ppt formed. (No ppt is formed by NG, PA, NGc, NGu, NC, EDNA, Ednatol, BkPdr and Pentolite)

Test 2: Place 0.05g of unknown material in an indenture of a wh porcelain spot test plate. Add 2–3 drops of a 65–68% aq soln of ethylenediamine, and stir. Note the color of the soln (not the solid). (NG produces no coloration, nor does NGc, NGu, EDNA, NC, and BkPdr)

Test 3: Same as Test 2, but add 2–3 drops of DPhA reagent (1g DPhA in 100ml concd sulfuric acid). Stir and observe the color after 1 min. (Deep blue is produced by NG, NGc, Ednatol, & Tetrytol; blue isproduced by Tetryl, EDNA, NG, NC and BkPdr)

Test 4: Same as Test 2, but add an equal amt of cryst thymol and 3 drops of concd sulfuric acid. Stir and observe the color after 5 mins. (NG gives green color and so does Tetryl, NGu, AN, PETN, Tetrytol, Amatol, Ammonal, Pentolite and BkPdr)

All this can mean that, if the aq soln of unknown sample is colorless in Test 2, no ppt with Nessler's reagent, no color with ethylenediamine in Test 2, deep blue color with DPhA in Test 3 and green color with thymol soln in Test 4, it could be NG

Munch et al revised in Ref 13 the variety of spot color tests for the nitrate radical which can be used for detn of NG

Other methods for detn of NG are described in Refs 1, 2, 3, 4, 7, 8, 10, 10a, 11, 14, 15 & 16

The stability of NG is normally checked by Abel Heat Test which is briefly described in Vol 1 of Encycl, p A2-L. The test conducted accdg to Spec MIL-N-246B is described below. Since the test is sensitive to the amt of w in NG, it is required to test each sample for moisture content

Nitroglycerol for Use in Propellants, Requirements and Tests of Specification MIL-N-246B, 19 Feb, 1962.

3. Requirements:

3.1. Nitroglycerol. There are two types of NG used in proplnts: Type I is prepd from Grade B glycerol and Type II from partially polymerized glycerol. Grade B glycerol shall comply with Federal Specification O-G-491. Partially polymerized glycerol shall contain 27 to 31% by wt of polymer glycerol, expressed as diglycerol

3.2. Moisture Content. 0.5% (max) when detd as specified in 4.3.1. When NG is used at the point of manuf in the production of proplnt by the water-slurry method or by the water emulsion method, the moisture requirement shall not apply

3.3. Acidity or Alkalinity. The acidity as sulfuric acid or alkalinity as Na carbonate shall be 0.002% (max), when detd as in 4.3.2

3.4.1. Nitrogen Content for Type I. 18.40% (min) when detd as in 4.3.3

3.4.2. Nitrogen Content for Type II. 17.90% (min) when detd as in 4.3.3

3.5. Stability. When subjected to 82.2°C heat test as described in 4.3.4, NG shall not change the color of the std KI-Starch paper in less than 10 mins

4.2. Inspection and Provision. See p 4 of Spec

4.2.3.1. Sampling. Using a rubber dipper, a sample of approx 2 oz shall be removed from each lot and transferred to a rubber bottle with a rubber stopper. The bottle shall be labeled to show source of manuf, plant, contract or purchase order No, lot No and number of pounds in the lot

After disposal of the NG from the rubber sample bottle, the bottle shall be cleansed thoroughly with acetone, flushed during about 3 mins under direct flow of tap w, rinsed with distd w and dried

4.3. Test Methods and Procedures:

4.3.1. Determination of Moisture in NG by Karl Fischer Reagent. Place a 5–10g sample of the NG into a tared narrow-necked stopperable flask and retare. Titratedirectly with standardized Karl Fischer Reagent until a brown color remains for 30 sec, or use a potentiometric endpoint. Then,

$$\% \text{ Moisture} = \frac{100\,(KF)}{W}, \text{ where}$$

K = ml of reagent used, F its factor (g of w per ml of reagent) and W = wt of NG sample in g (More detailed description of Karl Fischer Method, including prepn of the reagent, is given in Vol 5 of Encycl, pp D1622-L to D1628-L)

4.3.2. Determination of Acidity or Alkalinity of NG. Place a 10g sample of the NG by pipet into a tared beaker and retare. Add 100ml of benzene and transfer to a 250-ml separatory funnel when dissolved. Wash twice with 50-ml portions of neutral distd w, combining the washes in a 250-ml beaker. Add several drops of bromothymol blue indicator and immediately titrate with 0.01N NaOH if yellow, or with 0.01N sulfuric acid if blue. Run a blank on 100ml of benzene and correct the first titration value. Then, % acidity (as sulfuric acid) or alkalinity (as sodium carbonate) = kVN/W, where k is 4.90 for acid or 5.3 for alkali titrant, V is corrected titrant vol in ml, N is actual normality of titrant, and W is weight of NG

4.3.3. Determination of Nitrogen in NG

4.3.3.1. Standardization of Nitrometer. Standardization of the DuPont 5-part form of Lunge nitrometer shall be made by Method 209.3 of Standard MIL-STD-286. A detailed description of the method as was conducted during WWII at Keystone Ordnance Works, Meadville, Pa is given in Vol 1 of Encycl (Ref 10a, pp A374-R to A376-L). The apparatus, illustrated on p A374-L, shall be used while following the description of standardization and of the procedure 4.3.3.3

4.3.3.2. Drying of Filter Paper. Whatman No 42 or equivalent filter paper shall be transferred to a glass weighing dish equipped with a glass cover. The uncovered dish and contents shall be heated at 100±2° for 2 hrs, the dish covered, and cooled in a desiccator

4.3.3.3. Procedure with Nitrometer. Quickly place at least four dried filter papers in a dry conical funnel, then immediately add about 10g of sample NG, apply suction and collect the NG in a dry flask. Place 0.70–0.75g of the filtered NG into a tared 25-ml beaker and retare. Add 5ml of glacial acetic acid and stir with a small glass rod to dissolve the NG. Using the rod to avoid spillage, transfer the soln quickly to the cup (a) of the generating (or reaction) bulb (E) of Fig reproduced here. Draw into the bulb without allowing air to enter. Rinse the beaker and rod with successive 5-ml portions of 25ml of 94.5±0.5% sulfuric acid, separately placing each wash into the bulb cup (a), agitating with the rod, and then drawing into the bulb (E). Force out any air inadvertently admitted to the bulb and close the upper stopcock (c) when the last of the wash has been added. Leaving the

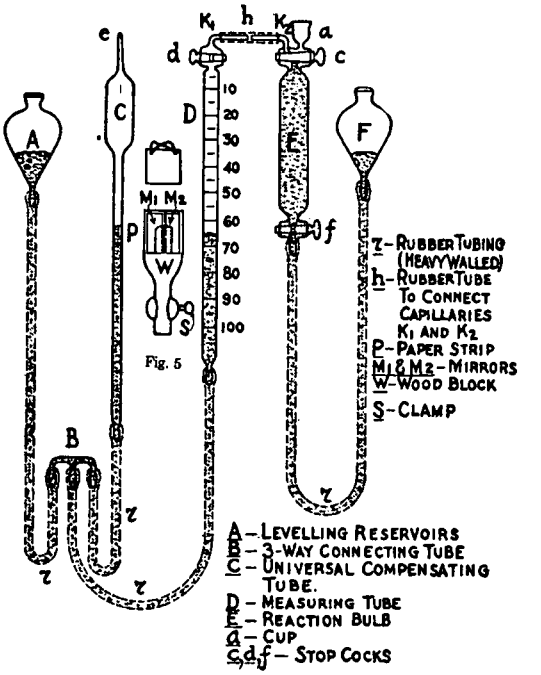


Fig. 5

The assembled apparatus is shown in Fig. It is mounted on a special iron stand which permits the adjusting of the various parts to the desired position

lower stopcock (f) open, adjust the level of the Hg in the reservoir (F) to 13–15 inches below the level of the Hg in the bulb (E). At this point inspect the cock (c) to see if there is any leak into (E). Make sure that cock (F) is open

Put on a full-view mask, wrap bulb (E) in several layers of cloth toweling and remove E from the rack. Gently shake the bulb, keeping the lower end fixed, until most of the NO has been generated. Then adjust the Hg reservoir (F) so that the Hg in the bulb drops almost to the lower shoulder, close the lower stopcock (f), and vigorously shake the bulb for 3 mins. Replace the bulb on the rack and let stand for 3 mins. Repeat the 3-min vigorous shaking and standing two more times. Care must be taken not to shake the bulb so vigorously that the acid is carried down thru (f) into the connecting rubber tubing (r)

Remove the towelling from (E) and raise it so that the Hg could flow thru (f) into (F), thus creating a vacuum in (E). When 1–2 inches of Hg (not of emulsion) is left in the bulb (E), close the stopcock (f)

With the cocks (c) & (f) tightly closed and held by the fingers, place (E) in a nearly horizontal position and shake vigorously for 2 mins.

By this time the mass must be a nearly homogeneous emulsion. Replace (E) on the rack and allow the emulsion to settle

While waiting, slightly grease the outside of capillaries K_1 & K_2 and see that they are filled with Hg. Raise or lower the measuring tube (D) so that (K_1) will be on the same level as (K_2). Slip a piece of heavy rubber tubing (h) 7 inches over (K_1) and push it all the way thru. Place the end of (K_2) against (K_1) and push the tubing (h) over the junction until both capillaries are equally covered

Now lower Hg reservoir (A), raise reservoir (F), and by opening the cock (f), let the Hg enter bulb (E) from (F) with formation of some pressure. Open the cock (d) of (D) and examine for any leakage. Normally the Hg in capillary will move slightly due to the presence of rubber connections and traces of air

If there is no leak, leave (d) open and, by manipulating carefully with (c), allow the NO gas to flow slowly from (E) to (D). Towards the end of transfer, partly close (c) to slow down the rate of flow and, just as soon as the acid starts to fill the capillaries, close (c). No acid shall enter (D) and no gas shall remain in the capillaries. Close (d), but leave (f) open

Adjust the levels of Hg measuring (D) and compensating (C) tubes until they are about the same, with that in the latter tube near the standardization mark. Let stand 20 mins to permit temp equilibration. Keep the Hg level in the compensating tube at the standardization mark and equalize the level in the measuring tube with the aid of a U-tube leveling device contg alc colored with methyl red indicator. Then, %N = A/0.9983W, where A is the reading of the Hg level on the measuring tube, W is the weight of NG used, and 0.9983 is a factor based upon the experimental detd average moisture content of NG filtered as described

4.3.4. Determination of Stability of NG. Filter a sample of the NG thru two layers of S&S No 604 paper (or equiv). Place 2ml of the filtered sample by pipet into each of three 5.5 x 0.5-inch test tubes in such a manner as to leave no NG on the sides. Stopper each tube tightly with a new cork, itself tightly attached to a glass rod, to the end of which in the tube is affixed a Pt holder with a 1 x 3/8-inch strip of standard KI-Starch indicator paper (attached by means of a forceps, not the fingers, and having a horizontal band in the upper half just wet thru with 50–50 (v–v) glycerol-distd w. Similarly equip a clean, dry tube as a blank. Adjust a bath to 82.2±1°, then submerse the four tubes to a depth of 2 inches and note the time of insertion. Against a white background lit by bright diffused daylight, observe the line demarking the bottom of the wet band from the dry paper (which line should be 3 inches above the NG, and an equivalent height above the bottom of the blank tube). To the nearest min note the elapsed time when any discoloration of the line in any of the NG tubes exceeds any discoloration of the line in the blank. Record this time as the value for the test, and do not average the values for three tubes

Nitroglycerol, Tests Not Required by Spec MIL-N-246B.

Determination of Purity of NG by Redox Titration. The two equations representing this method are: $NG + 9Fe(II) + 9H^+ = G + 9Fe(III) + 3NO + 3H_2O$, and $Ti(III) + Fe(III) + Ti(IV) + Fe(II)$. Excess ferrous ion in hydrochloric acid soln is used to reduce the NG to glycerol (G) and nitric oxide. Thiocyanate indicator denotes the presence of ferric ion during a titration of the latter with standard titanous ion, the blood-red color disappearing with the last of the ferric species. Air must be rigorously excluded from the system because of the ease of oxidation of Ti(III), and carbon dioxide is generally used because of its heavier than air feature. For the indicator dissolve 20g reagent grade ammonium thiocyanate in 100cc distd w, and filter if necessary. At the proper time transfer 5cc of this with a pipet whose tip has been removed for fast delivery. For the ferrous soln dilute 140g of ferrous chloride tetrahydrate and 50cc of concd HCl to 1 liter with w using deaerated acid and w, and mixing with a current of carbon dioxide (about 0.7N). For the titanous soln dilute 150cc of a commercial 20% titanous chloride soln and 100cc concd HCl to 1 liter with w (about 0.2N); protect from air and store out of contact with light. Standardize the Ti(III) against Fe(III) (prepd by oxidizing standard ferrous ammonium sulfate (solid) with $KClO_3$, destroying excess chlorate by evaporating to dryness:
$6Fe(II) + KClO_3 + 6HCl = 6Fe(III) + KCl + 3H_2O$
and $KClO_3 + 6HCl = KCl + 3H_2O + 3Cl_2$. Place about 2g NG in a tared beaker and retare. Dissolve in carbon dioxide-free HAc, and transfer

quantitatively to a 250-cc volumetric, and dilute to the mark with HAc, mixing well and avoiding any temp change before use. Displace the air in a 300-cc Florence flask (equipped with a side arm for carbon dioxide) for 5 mins. Place a 25-cc portion of the NG–HAc soln in the flask followed by 15cc of the Fe(II) soln, and then 25cc of 15% (w/w) HCl, plus a few boiling chips. Connect to a reflux condenser and boil gently for 5 mins on a hot plate. The color will change from yel to dark green to reddish brown. Increase the flow of carbon dioxide and immerse the flask in a large beaker of cold w, lightly blocking the top of the condenser until all vapors have condensed. Once cooled to RT, remove the condenser, insert a rubber stopper with a piece of glass tubing, and titrate with the Ti(III) – the carbon dioxide exiting thru the annular space between the buret tip and the tubing. As soon as the reddish color disappears, add the 5cc of indicator and continue the titration until the red color disappears. Then, % NG = 2.523VN/0.1W, where V is the vol of Ti(III) corrected for a blank, N is the normality of the Ti(III), and W is the weight of the NG sample. Variations of this redox titration may be found in Refs 3, 4 & 8

Determination of Purity of NG by the Dewarda Method. This method involves the saponification of NG to glycerol and nitrate ion. The latter is then reduced with Dewarda's alloy, a mixture of Al, Zn and Cu. The resultant ammonia is then distd out and titrated (Refs 2, 5, 10 & 17)

Determination of NG by Gas Liquid Chromatography. This technique is useful for measuring NG or other polyol nitrates present in air. Bubble a 10-liter sample thru two 10-ml containers of alc, then inject some of the alc soln onto a siliconized Chromosorb P column coated with 10% Igepal CO-880. Run the analysis at 160° under nitrogen carrier to detect a min of 2mg (Ref 14)

Determination of Composition of Mixed Acid. This analysis consists of titration of a sample for total acid, heating another sample to drive off the nitric acid and titrating the remaining sulfuric, and titrating a third sample with permanganate to determine the nitrosylsulfuric acid content. The latter is subtracted from the values for the nitric and sulfuric, the three corrected values added, and the (positive) difference from 100 taken to be the amount of w (See Ref 16 for details)

Refs on Analysis of NG: 1) Marshall **2**(1917), pp 739–40 (Estimation of small quantities by reaction with phenol disulfonic acid to form PA and colorimetric measurement against K nitrate standards) 2) Koehler et al, AnnChimAnal-ChimAppl **2**, 271–2(1920) & CA **14**, 3794(1920) (Devarda method) 3) W.W. Becker, IECAnalEd **5**, 152–4(1933) & CA **27**, 2909(1933)(Redox method for NG and NGc in the presence of DNT) 4) H. Shankster & T.H. Wilde, JSocChemInd **57**, 91–2(1938) & CA **32**, 4471(1938)(Reverse Redox method) 5) R. Vandoni, MP **28**, 136–41 (1938) & CA **33**, 7695(1939)(Devarda method) 6) Ibid **29**, 204–5(1939) & CA **34**, 1850(1940) (Influence of moisture on the Abel Heat Test at 80°) 7) V.G. Gurevich et al, Zavodskaya-Lab **12**, 553–4(1946) & CA **41**, 1954(1947)(Determination of small quantities of NG in the air in the presence of nitrogen oxides)(Involves saponification to nitrite which is determined colorimetrically) 8) P. Fainer, CanJChem **29**, 46–53(1951) & CA **45**, 6156(1951)(Reverse Redox method) 9) M. Halse, MeddNorskFarm-Selskap **16**, 166–9(1954) & CA **49**, 13024(1955) (Determination of NG in mixtures with PETN) (Saponification to nitrite at 20°, then reaction with sulfanilic acid and α-naphthyl amine for a colorimetric measurement against nitrite standards) 10) P. Lhoste, MP **37**, 149–52(1955) & CA **50**, 17451(1956)(Rapid analysis of NG tablets) (Dewarda method) 10a) G.C. Whitnack et al, AnalChem **27**, 899–901(1955)(Polarographic detn of NG in double-base proplnts) 10b) J. Lamond, Analyst **82**, 768–69(1957) & CA **52**, 3345(1958)(The addn of equiv vol of Nessler's reagent to an alc soln contg 1% NG results in a black ppt which is Hg. Testing directly in proplnts might produce color masking the ppt. For this reason it is advisable to test the extract of NG with 4/1–EtOH/H_2O) 10c) Encycl of Expls, PATR **2700**, Vol 1(1960), pp A373–A378(Detn of nitrogen content by means of duPont's nitrometer) 11) I.M. Kolthoff & P.J. Elving, Edits, Treatise on Analytical Chemistry, Part II, Vol **5**, J. Wiley-Interscience, NY (1961), pp 299–303 (A.J. Clear & M. Roth, Nitrogen–duPont nitrometer) 12) MIL-N-246B (1962) (Nitroglycerin) 12a) StdMethodsChemAnalysis, Vol **2**, Part **B** (1963), 1335–36(NG. Determination of moisture by desiccation and by Karl Fischer reagent. Detn of acidity and alkalinity. Percentage of nitrogen by nitrometer. Stability test with KI-

starch paper at 82.2°C); 1347–48 (Detection of NG by colorimetric methods) 13) J.C. Munch et al, JPharmSci **53**(7), 842–3(1964) & CA **61**, 11333(1964)(Sensitivity of color tests for nitrites, nitrates, and NG. I. Solutions in distilled water) 14) E. Camera & D. Pravisani, AnalChem **39**(13), 1645–6(1967) & CA **67**, 111212(1967)(Determination of alkyl polynitrates by electron capture gas chromatography – application to air pollution) 15) J. Vich et al, Explosivst **18**(2), 42–8(1970) & CA **73**, 57623(1970)(Chromatographic analysis of nitric esters)(Discusses recent methodd for separating NG, EGDN, DEGDN, PETN and compares paper chromatography, thin layer chromatography, and spectrophotometry) 16) F.D. Snell & L.S. Ettre, Eds, "Encyclopedia of Industrial Chemical Analysis", Vol **16**, J. Wiley-Interscience, NY (1972), pp 498–500(F.A. Lowenheim, "Nitrogen Compounds-Analysis of Mixed Acid" 17) Ibid, p 467 (Devarda's Method)

Glycerol Trinitrite (Glycerintrinitrit, in Ger), $ONOCH(CH_2ONO)_2$; mw 179.06, N 23.46%, OB to CO_2 –22.3%; yel liq (sl impure), sp gr 1.291 at 10/15.5°, bp 150°(sl dec); sol in eth, chlf & benz; immisc with w; dec in air, w & abs alc. Prepd by treating cold glycerol with N_2O_5
Refs: 1) Beil **1**, 514 2) O. Masson, Ber **16**, 1697(1883)

Nitroglycerol Shell was invented by Fannon-Winslow about 1885. It contained two glass jars, one holding glycerol 1p and concd sulfuric acid 1p; the other nitric acid 2p and sulfuric 1p. A time fuze exploded a small charge of BkPdr which broke the bottles, mixing the contents and producing NG. The impact of the shell exploded the NG causing the shell to burst
Note: More successful than NG shells were shells contg NG gelatinized with 7–10% of CC (Collodion Cotton) to which was added about 4% camphor (Ref 2, p D1610-L). Other types were loaded with Dynamite. They were propelled by compressed air from special pneumatic cannons. Accdg to Ref 1, p 934, the first of such shells was built in 1883 by Mefford of Ohio (See also Ref 2, p D1610-L). Accdg to Pérez Ara (1945), pp 333–34, Americans used large caliber pneumatic cannons in their coastal defenses and during Spanish-American War for bombarding Havana (See Ref 2, p D1610-R)
Refs: 1) Van Gelder & Schlatter (1927), pp 933–34 2) Encycl of Expls, Vol **5**(1972), pp D1609 to D1610

Glycerolpentaerythritol Ether Pentanitrate, $O_2N.O.CH_2CH(ONO_2).CH_2.O.CH_2.C(CH_2ONO_2)_3$; mw 435.21, N 16.09%, OB to CO_2 –23.9%; crysts, mp 54.5–55°, sp gr 1.57 at 20/20°, n_D 1.531 and 1.520 at 31°. No prepn is given in Ref 2; presumably a mixed acid nitration at 0° was performed on the (freshly made) parent
Refs: 1) Beil, not found 2) R. Evans & J.A. Gallaghan, JACS **75**, 1249(1953) & CA **49**, 3811(1955)

Glycerol-Peroxide Explosive consisting of glycerol, w & hydrogen peroxide in the ratio within the triangle defined on a trinagular graph by the corners: glycerol 5, w 10 & peroxide 85%; glycerol 15, w 52 & peroxide 33%; and blycerol 65, w 0 & peroxide 35%. Such compns are insensitive to mechanical shock, including rifle bullet, but are detonated by a blasting cap
Ref: E.S. Shanley & H.O. Kauffmann, USP 2452074(1948) & CA **43**, 1190(1949)

Glycerol Substitutes. During WWII, the Germans developed several processes for preparing either glycerol or compds with similar properties. The so-called Oppau Process of IGFarbenindustrie AG involving propylene chlorination and hydrogenation produced pure glycerol, while other processes gave mixts of glycerol with other substanves

One such mixture was *Glycerogen* (qv)

Glycerol-Sugar Nitrates, including **Nitrohydrene.** As sugar (saccharose $C_{12}H_{22}O_{11}$) is less expensive and more abundant, especially during a war, its nitration has been attempted. The resulting Nitrosugar (NSu), usually *Heptanitrate*, $C_{12}H_{15}O_4(ONO_2)_7$, proved to be a very powerful expl, but very unstable because of the presence of impurities. Better results were obtd

by nitrating a 20–25% sugar soln in glycerol. The resulting expls were nearly as powerful as straight NG, but less stable. In order to increase their stabilities (especially at ca 50°C and above), 0.1–0.2% of DPhA was added (Refs 1, 2 & 3)

The idea of prepg such mixts originated with Björkmann who in 1880 nitrated (in Sweden) a soln of glucose 25 parts in glycerol 75ps. Then in 1911 an EnglP 17221 was issued to Cocking & Kynoch Ltd protecting a process of nitrating any mixt of glycerol and sugars, and finally a USP 1149487 was issued in 1915. Before this, however, the DuPont Co started to nitrate a soln of 20 parts of sugar in 80ps of glycerol and named it *Nitrohydrene*. Accdg to Symmes (See translator's footnote 58 in Ref 1, p 251), the ordinary table grade of refined sugar was used in the US, but a small quantity of refined glucose was added to prevent crystallizing NSu when shipped during cold weather

Description of nitration process and some properties of NG–NSu mixts are given by Naoúm (Ref 1); Davis (Ref 2) lists the same props as Naoúm. If the expl strength of NG by Trauzl Test is 550cc, then the 80/20 Nitrohydrene gives 533 and a 75/25 mixt 514cc

Accdg to Pérez Ara (Ref 4), Björkmann gave the name of *Glukodin* to the mixt of NG 91.18 & NSu 8.82%, prepd by nitrating a soln of saccharose in NG. Björkmann also proposed Dynamites contg Glukodin, NC, Na nitrate & sugar. Daniel (1902), p 345 gave credit to Dittmar for using Glykodin in two expl mixtures

Giua (Ref 5) lists the following Dynamites contg NG–NSu:

a) NG 33.2, NSu 3.2, NC 24.3, Na nitrate 31.3 & sugar 8.0%; and

b) NG 30.2, NSu 4.0, NC 19.2, Na nitrate 37.8 & sugar 8.7%. They are similar to Dynamites listed in Pérez Ara and Daniel

In Germany, the scarcity of glycerol during WWI led to the use of NG–NSu mixts in mining expls, but this practice was discontinued in peace time because saving in NG did not amount to more than 10–12%, but the acid consumption was greater than in nitration of glycerol

Refs: 1) Naoúm, NG (1928), 250–55 2) Davis (1943), 239–40 3) Clift & Fedoroff **2**(1943), pp G2 & G3 4) Pérez Ara (1945), 352 5) Giua, Trattato **VI**(1)(1959), 345

Glycerol (or Glyceryl) Trilactate, $HO.CH_2.C(O)O.CH[CH_2.O.C(O)CH(OH).CH_3]_2$; mw 308.29; liq, does not boil at 40° at 0.4mm Hg; sol in w, alc & methanol. Prepd by hydrogen reduction in the presence of Pd of the corresponding tris-o-benzyl compd, the benzyl having been added prior to the ester formation for protection. Apparently the compd is too unstable towards heat for bp determination (See Ref 3)

Refs: 1) Beil, not found 2) L. Feldmann & H.O.L. Fischer, ArchBiochem **14**, 117–24 (1947) & CA **42**, 1884(1948) 3) P.E. Brandt et al, ActaChemScand **22**(5), 1691–3(1968) & CA **69**, 111308(1968)(Gas chromatographic separation as tris-trimethyl silyl ether)

Glycerol (or Glyceryl) Trinitramine (NGX) or **1,2,3-Trinitraminopropane.** It will be described under Triaminopropane and Derivatives

Glycerol-α-[2,4,6-Trinitrophenylether] Dinitrate; *1-[2′,4′,6′-Trinitrophenoxy]-propane-2,3-dinitrate,* **Trinitril** or **Dinitroglycerol Picrate** [Called in Colver (Ref 5) *α-Trinitrophenol-dinitroglycerin*], $(O_2NO)CH_2.CH(ONO_2).CH_2.O.C_6H_2(NO_2)_3$; mw 393.18, N 17.81%, OB to CO_2 –34.8%; crystals, mp 128.5°, begins to decomp at 119–20°. Was first prepd by Voswinkel (Ref 2) in 1893 by treating an alcoholic soln of PA salts with a chloro- or bromodinitrohydrin, followed by crystallization of the resulting product. Blatt (Ref 7) lists three methods of prepn

It is an expl sl more powerful than Tetryl with nearly the same sensitivity to friction but more sensitive to impact

Following properties are listed in Blatt (Ref 7) & ADL (Ref 8):

Impact by drop of 10kg wt. 7–10cm vs 23–24 for Tetryl;

Initiation Sensitivity. Detonates by No 6 cap;

Power by Trauzl Test. 128–130% PA or 147% TNT;

Rate of Detonation 3200m/sec, loosely loaded in a Cu shell;

Rifle Bullet Test. Same as for Tetryl;

Stability. Explodes at 235° when heated at the rate of 5°/min;

Surveillance (Storage) at 80°. Lost in 8 days 28% wt and developed acid reaction

It is not as good an initiator for PA as is Tetryl (Ref 7)

Lewis (Ref 6) proposed it as a component for detonator and booster charges

Refs: 1) Beil, not found 2) A. Voswinkel, GerP 74293(1893) & SS **9**, 194(1893) 3) Zentralstelle für wissensch-techn Untersuchungen in Neubabelsberg, Annual Rept of 1909 4) Dynamit Nobel, BritP 24352 & CA **10**, 1597 (1916) 5) Colver (1918), 699 6) H.A. Lewis, USP 1560426 & CA **20**, 112(1926) 7) Blatt, OSRD **2014**(1944) 8) ADL, PureExplCompds, Part 1(1947), 124 & 148

Glycerol (or Glyceryl) Tripermanganate, $O_3MnOCH(CH_2OMnO_3)_2$; mw 397.88, OB to CO_2 & MnO_2 −10.0%. There is no reference to this compound in the open literature. The inventor, Mr Emil Dvorak, claimed that the compd may be prepd by the reaction between silver permanganate & trichlorohydrine. It was proposed as an expl possibly suitable for military & commercial uses. All attempts to prep this compd at Picatinny Arsenal were unsuccessful (Ref 3)

Refs: 1) Beil, not found 2) CA, not found 3) O.E. Burton, "Study of the Preparation and Explosive Characteristics of Glyceryl Permanganate", PATR **124**(1931)

Glycerol (or Glyceryl)-tris(Trinitrobutyrate), $(NO_2)_3C.CH_2.CH_2.C(O)O.CH[CH_2O.OC.CH_2.CH_2.C(NO_2)_3]_2$; mw 707.35, N 17.82%, OB to CO_2 −32.8%; crysts, mp 92–3°. There is no reference to this compd in the open literature. Accdg to Ref 3 below, the material flashes on a hot plate, and detonates with a hammer blow

Refs: 1) Beil, not found 2) CA, not found 3) USRubberCo Quarterly Prog Rept **19** (1 May–1 Aug 1952), p 5 (Contract NOrd 10129)

Glyceronitre. Same as Fortis described in this Vol, p F174-R to F175-L and in Daniel (1902), 304 & 355

Glyceronitrocellulose. See Glyceropyroxyline

Glyceropyroxyline. One of the Clarke's Propellants, listed in Vol **3** of Encycl, p C327-L and in Daniel (1902), p 355

Glyceryl or Propenyl. Trivalent radical: $-H_2C.CH.CH_2-$, derived from glycerol, which may be called glyceryl hydroxide

Glyceryl Triacetate. See Tracetin on p A31-R of Vol **1**, under ACETINS

Glycide or Epoxy-1-propanol (γ-Oxypropylenoxyd, in Ger)(*Glycidol, Epihydric Alcohol* or *Anhydride of Glycerol*), $O.CH_2.CH.CH_2OH$; mw 74.08, colorless liq, sp gr 1.114 at 16/16°; bp 62° at 15mm and 166–67° at 760mm (with sl decompn); very sol in w & in alcl sol in eth & benz; sl sol in petr eth & xylene. Can be prepd by treating glycerol-α-monochlorohydrin with calcd amt of K or Na ethylate (Ref 3)

Although glycide is supposed to be the parent compd of Nitroglycide, it is not used for such purpose because the oxygen bond breaks down on action of strong acids. On the other hand, weak nitric does not break the O bond, but transforms glycide into glycerol mononitrate (Refs 2 & 4). For this reason Nitroglycide is prepd by an indirect method described below

Refs: 1) Beil **17**, 104, (50) & [104] 2) M. Hanriot, AnnChimPhys(Paris) (5), **17**, 118(1879) 3) J.U. Nef, Annalen **335**, 232(1904) 4) Naoúm, NG (1928), 171 5) Vol **5** of Encycl, p E106-R (Brief description as 2,3-Epoxy-1-propanol)

Glycide Nitrate or Nitroglycide (Nitrate of Epihydric Alcohol), $O.CH_2.CH.CH_2(ONO_2)$; mw 119.08, N 11.76%, OB to CO_2 −60.5%; colorless mobile liq, very volatile at ord temp, sp gr 1.332 at 20°/4, fr p − did not freeze at −20°; bp 94° at 20mm or 174–75° at 760mm (with partial decompn), detonates at 195–200°(Ref 4); sol in w (5% at 20°); miscible with alc, eth, acetone, et acet & NG; decompd by hot w. Reacts explosively on contact eith concd sulfuric

acid. Reacts with concd nitric acid with great evoln of heat and the formation of NG and Glycerol Dinitrate

Can be prepd by shaking glycerol dinitrate (both alpha and beta isomers) at RT with 30% aq soln of Na or K hydroxide as was done by Naoúm in 1907 (Refs 3 & 4). The Nitroglycide deposited as a clear oil, was isolated, washed with w and dried in a desiccator. The yield was practically theoretical

Nitroglycide is a HE, very brisant (72% that of NG), about 52% as powerful as NG (compared to 36% for mononitroglycerol, whose anhydride it is), much less sensitive than NG: 10–20cm vs 2cm for NG with 2kg wt. It is more sensitive to initiation by an ordinary Blasting Cap than NG, probably (in the opinion of Naoúm) due to the lower viscosity of Nitroglycide

Recommended as a good plasticizer for NC (Ref 5)

Refs: 1) Beil **17**, 106 & [105] 2) J.U. Nef, Ann **335**, 238(1904) 3) Naoúm, NG(1928), 175 4) Davis (1943), 216–19 5) R.W. Van Dolah, USNavalOrdnLab MemoRept **10068** (1949), p 24

Glycine. See Aminoacetic or Aminoethanoic Acid in Vol 1 of Encycl, p A178-L

Glycine Nitrate. See Aminoacetic Acid Nitrate in Vol 1, p A178-L

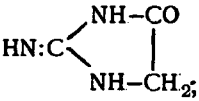
Glycine, Nitro. See Nitroaminoacetic Acid or Nitroglycine in Vol 1, p A178-R

Glycinonitrile, N-(4,6-diamino-s-triazin-2-yl), $C_5H_7N_7$; mw 165.15, N 59.39%; crysts (+2w), mp 220–5°; sol in w, alc & dil acids; insol in hydrocarbons. Prepd from one mole of the 4,6-dichloro-compd and five moles of concd ammonia

Refs: 1) Beil, not found 2) I. Hechenbleikner, USP 2476546(1949) & CA **44**, 4518(1950)

Glycocoll. See Aminoacetic Acid in Vol 1 of Encycl, p A178-L

Glycyamidine or Glycolylguanidine,



$HN{:}C(NH{-}CO)NH{-}CH_2$; mw 99.09, N 42.41%; colorless crysts, mp – browns at 220°; v sl sol in water; insol in alc & eth. Can be prepd by dehydration of glycocyamine and by other methods. Forms numerous cryst salts (Ref 1) with metals & org compds

Refs: 1) Beil **24**, 244, (287) & [127] 2) Hackh's Dict (1944), 383-L

NOTE: No azido or nitrated derivs were found in Beil

Glycocyamine or Guanylglycine, $HN{:}C(NH_2).NH.CH_2.CO_2H$; mw 117.11, N 35.88%; colorless ndls (from w), mp 270–80°; sl sol in w; v sl sol in alc or ether. Can be prepd from cyanamide & glycine. Gives on dehydration glycocyamidine

Refs: 1) Beil **4**, 359, (477), [793] & {1165} 2) Hackh's Dict (1944), 383-L

NOTE: No azido or nitrated derivs were found in Beil

GLYCOLS (Gc's) AND DERIVATIVES (Diols or Dihydric Alcoholx)

Glycols are alcohols of the aliphatic series that have two hydroxyl (–OH) groups attached to separate carbon atoms. They may be subdivided into (Ref 2):

Diprimary–	contg two	$-CH_2OH$ groups
Disecondary –	" "	=CHOH "
Ditertiary –	" "	≡C.OH "
Primary-secondary	" one	$-CH_2OH$ and
	"	=CHOH groups
Primary-tertiary –	" "	$-CH_2OH$ and
	"	≡C.OH groups
Secondary-tertiary –	" "	=CHOH and
	"	≡C.OH groups

or they may be subdivided accdg to the number of carbon atoms they contain, such as C_2 glycols, C_3 glycols, C_4 glycols etc (Ref 3). Accdg to the Geneva Nomenclature, the C_2 glycol is called "ethanediol", the C_3 "propanediol" etc, but in more common use are the names ethyleneglycol,

propyleneglycol etc. For polymerized glycols, known in general as polyglycols, the names diethyleneglycol, triethyleneglycol, or polyethyleneglycol are used for derivatives of ethyleneglycol, or polypropyleneglycol for derivatives of propyleneglycol

In the case of polyglycols of high molecular weight, some manufacturers attach numbers to their trade names, eg, the Union Carbide Co uses such names as Carbowax 1540, Carbowax 4000 etc for their solid polyethyleneglycols. The Dow Chemical Co calls their products: Polyglycol E400, E600, E1200 and E4000

Polyglycols were known in Germany as Polyglykol P-6, P-8, P-9, P-12, P-60 and P-80, where P-6 etc refers to the units of ethylene oxide used in the manufacture

Glycols have been known since 1859, when French chemist Wurtz (pronounced Würtz) prepd ethyleneglycol (See in this Vol, p E247-R). The first polyglycol was prepd by Lourenco in 1859 (See Diethyleneglycol in Vol **5**, pp D1231ff)

Curme and Johnston (Ref 10, pp 7–10) gave a fairly complete history of the development and use of glycols in Germany and the USA. A short historical background is given in this Vol under "Ethyleneglycol" and under other individual glycols

The prepn and props of the following glycols and their derivs are given by Curme and Johnston (pp 4–5), but only about 50% of these compds are of interest from the Ordnance viewpoint:

List of Glycols:

Ethyleneglycol, $HOCH_2.CH_2OH$;
Propyleneglycol, $CH_3.CHOH.CH_2OH$;
Trimethyleneglycol, $HOCH_2.CH_2.CH_2OH$;
1,3-butanediol, $CH_3.CHOH.CH_2.CH_2OH$;
1,4-butanediol, $HOCH_2.CH_2.CH_2.CH_2OH$;
2,3-butanediol, $CH_3(CHOH)_2CH_3$;
2-butene-1,4-diol (trans-form), $HOCH_2.CH{:}CH.CH_2OH$
2-butyne-1,4-diol, $HOCH_2.C{:}C.CH_2OH$;
1,5-pentanediol, $HOCH_2(CH_2)_3CH_2OH$;
2,4-pentanediol, $CH_3.CHOH.CH_2.CHOH.CH_3$;
1,6-hexanediol, $HOCH_2(CH_2)_4.CH_2OH$;
2,5-hexanediol, $CH_3.CHOH(CH_2)_2.CHOH.CH_3$;
2-methyl-1,3-pentanediol,
$CH_3.CH_2.CHOH.CH(CH_3).CH_2OH$;
2-methyl-2,4-pentanediol,
$CH_3.CHOH.CH_2.C(CH_3)OH.CH_3$;
2,3-dimethyl-2,3-butanediol (pinacol),
$CH_3.C(CH_3)OH.C(CH_3)OH.CH_3$;
2,4-heptanediol, $CH_3.(CH_2)_2.CHOH.CH_2.CHOH.CH_3$;
2,2-diethyl-1,3-propanediol,
$HOCH_2.C(C_2H_5)_2.CH_2OH$;
2-ethyl-1,3-hexanediol,
$CH_3.(CH_2)_2CHOH.CH(C_2H_5).CH_2OH$;
2-ethyl-2-butyl-1,3-propanediol,
$HOCH_2.C(C_2H_5)(C_4H_9).CH_2OH$;
diethyleneglycol, $O(CH_2.CH_2OH)_2$;
triethyleneglycol, $HO.C_2H_4.O.C_2H_4.O.C_2H_4OH$;
tetraethyleneglycol,
$HO.C_2H_4.O.C_2H_4.O.C_2H_4.O.C_2H_4OH$
dipropyleneglycol, $O(CH_3CHOHCH_2)_2$

Note: In addition to the compds listed above, there is a substance called *methyleneglycol* (existing only in soln) which is not, strictly speaking, a glycol because the two hydroxyl groups are attached to the same carbon atom, $CH_2(OH)_2$

Methods of prepn have been reviewed by Lawrie (Ref 1), Naoúm (Ref 2), Ellis (Ref 4), Sherwood (Ref 9) and Curme & Johnston (Ref 10). Laboratory methods of prepn of ethyleneglycol are listed in Ref 10, pp 14–15. Methods of manuf are listed in Ref 10, pp 15–22, and in this Encycl under individual compds, such as diethyleneglycol, ethyleneglycol, propyleneglycol, triethyleneglycol etc

Physical properties of glycols. In the pure state, the simple glycols are stable, colorless liquids with freezing points below 0° and boiling points in the vicinity of 200°. The lower glycols (such as ethylene and propylene) are completely sol in w, but solubility decreases with increase in molecular weight. The lower members of the polyethylene- and propylene glycols are colorless, viscous liquids, while the higher molecular compds are wax-like solids. With increasing mw of the polyglycols, solubility in w and organic solvents as well as hygroscopicity decrease, while the freezing point, density, flash point and viscosity increase (Ref 10, pp 27–61 and 210–40)

Chemical properties of glycols. They are similar to those of aliphatic alcohols and the glycols may be oxidized (in the vapor phase and in the presence of catalysts) to the corresponding acids. They are easily esterified by inorganic and organic acids to form esters. Ethers (such as methyl, ethyl etc) may be prepd by treating

the glycols with alkyl sulfates inthe presence of alkali (See Ref 10, "Subject Index", pp 370–71 and 381, under "Reactions")

Toxicity. The glycols and their polymeric derivatives have relatively low toxicity, but their nitrates are toxic. This subject is discussed in Ref 10, pp 300–327, under the title: "Physiological Aspects of the Glycols". It includes 125 refs. The toxicity of individual glycols is described briefly under each compd but it might be said generally that they are more or less toxic when taken orally or when mists or hot vapors of them are inhaled in large quantities. They irritate the skin to a certain extent, but do not seem to be absorbed very readily thru it. However, long exposures over large areas of the skin should be avoided. Some of the glycols and polyglycols are considered as safe for use in cosmetics and in pharmaceuticals and, in the case of propylene glycol, in foodstuffs (Ref 10, pp 300–327)

Uses.

Glycols and their derivatives are used extensively in industry and the household. The simple glycols are used in antifreeze solns for automotive cooling systems, commercial refrigerators etc. As liquid coolants, they are used in aircraft engines, machine

Table

Single-Dose Oral Toxicities of Various Glycols and Related Compounds

Compound	LD_{50} for Rate in g/kg	Compound	LD_{50} for Rate in g/kg
Acetals	7.2	Ethyleneglycol	8.5
1,3-Butanediol	18.6	Ethyleneglycol Dinitrate	No data
1,4-Butanediol	No data	2-Ethyl-1,3-hexanediol	2.5
2,3-Butanediol	>5 (for rabbits)	Ethylene oxide	No data
Carbowax 1000	42	Glycolic acid	1.95
Carbowax 1500	44.2	Glyoxal	2.02
Carbowax 1540	51.2	2,5-Hexanediol	5.0
Carbowax 4000	59	2-Methyl-2,4-pentanediol	4.7
Carbowax 6000	>50	Polyethyleneglycol 200	34.0
Diethyleneglycol	20.8	Polyethyleneglycol 300	38.9
2,2-Diethyl-1,3-propanediol	No data	Polyethyleneglycol 400	43.6
Dimethyl dioxane	3.0	Polypropyleneglycol (85% di-, 15% triglycol)	14.1
2,3-Dimethyl-2,3-butanediol or Pinacol	No data	Polypropyleneglycol 425	2.91
Dioxane	About 6	Polypropyleneglycol 1025	2.15
Dipropyleneglycol	14.8	Polypropyleneglycol 2025	9.76
Ethers and esters of various glycols–See Curme & Johnston, pp 305, 306, 309, 311, 313, 322		Propyleneglycol	26.4
2-Ethyl-2-butyl-1,3-propanediol	3.0	Propylene oxide	1.14
Ethylene chlorhydrin (3.5g inhaled from the atmosphere killed a man)	0.095	Propylene oxide tetramer	1.8
		Triethyleneglycol	22.1
		Trimethyleneglycol (about twice as toxic to small animals as propyleneglycol)	

guns and Army tanks. Some derivatives of glycols are suitable gelatinizing agents for NC (See Ref 10, "Commercial Applications", pp 63–73 and 241–49)

Nitrated glycols, especially the lower members such as ethylene-, propylene-, triethylene-, and diethyleneglycol nitrates, are used in expl and proplnt compns. Nitrates of the higher glycols, or polyglycols, were proposed as ingredients of Diesel fuels in order to raise their "cetane number" (See Vol **2** of Encycl, p C145-R

Analysis and Test Methods.

Analytical procedures used in connection with glycols are described in the book by Curme and Johnston, pp 328–351 (54 refs are included) and under individual glycols in this Encycl

The following glycols are described in this Encycl:

Butyleneglycols or Butanediols and Derivatives are in Vol **2**, pp B368-R to B370-R;

Diethyleneglycol and Derivatives are in Vol **5**, pp D1231-R to D1233-L;

Dipropyleneglycol and Derivatives are in Vol **5**, p D1497-L & R;

Ethyleneglycol and Derivatives are in this Vol, pp E247-Rff

Trimethyleneglycol and Derivatives is described as Dimethylolmethane or Propanediol in Vol **5**, p D1355-L;

Propyleneglycol and Polyglycols will be described under P's;

Triethyleneglycol will be described under T's

Refs: (See also under individual Glycols, such as Diethyleneglycol, Dipropyleneglycol, Ethyleneglycol, Propyleneglycol, Triethyleneglycol, Trimethyleneglycol, Polyglycols)

1) J.W. Lawrie, "Glycerol and the Glycols–Production, Properties and Analyses", Chem Catalog Co (Reinhold Pubg Co), NY (1928) (ACS Monograph No 44), pp 361–95 2) P. Naoúm (translated from German by E.M. Summes), "Nitroglycerine and Nitroglycerine Explosives", Williams & Wilkins Co, Baltimore, Md (1928), pp 210–239 3) A.L. Bernouilli & W. Kambli, HelvChimActa **16**, 1187 (1933) 4) C. Ellis, "Chemistry of Petroleum Derivatives", Vol **1**, Chem Catalog Co, NY (1934) pp 506–11; Vol **2**, Reinhold, NY (1937), pp 532–35 4a) E. Browning, "Toxicity of Industrial Organic Solvents", HM Stationary Office, London (1937) 5) N.A. Milas & S. Sussman, JACS **59**, 2345 (1937) 5a) K.B. Lehmann & F. Flury, "Toxicology and Hygiene of Industrial Solvents", Williams & Wilkins, Baltimore, Md (1943) 6) J.F. Walker, "Formaldehyde", Reinhold, NY (1944) (ACS Monograph No 98), pp 29–30 & 140–42; and 3rd edition (1964), 96, 217, 267–67 & 635 7) Hackh's Dict (1944), 383 8) J.F.C. Gartshore, BIOS Final Report No **360** (1945) 9) P.W. Sherwood, Petroleum Refiner **28**(3), 129; (7) 120 (1949) 10) C.O. Curme, Jr & F. Johnston, "Glycols", Reinhold, NY (1952) (ACS Monograph No 114) (1370 refs) 11) R.E. Kirk & D.F. Othmer, Eds, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **10** (1966), pp 638–76 (M. Freifeld & E.V. Hort, "Glycols")

Addnl Refs for Glycols:

A) H. Moureau et al, MP **28**, 252–64 (1938) (Prepn of glycol starting from ethylenic hydrocarbons)

B) P. Aubertein, MP **30**, 7–42 (1948) & CA **45**, 353 (1951) (The following nitrated glycols were prepd after WWII at the Laboratoire Centrale des Poudres, Paris: Ethyleneglycoldinitrate; Diethyleneglycoldinitrate; Triethyleneglycoldinitrate; 1,3-Butanedioldinatrate; 1,4-Butanedioldinitrate and 2,3-Butanedioldinitrate)

C) P. Aubertein, MP **41**, 57–64 (1959) (Glycol and its nitrated derivatives)

Glycol Butylether Acetate. See Ethyleneglycol Butylether Acetate in this vol, p E254-L

Glycol Condensation Products. See under Ethyleneglycol Condensation Products in this vol, p E254-L

Glycol Dinitrate. See ETHYLENEGLYCOL DINITRATE in this vol, p E259-R

Glycol Dinitrate – Nitroisobutylglycol Dinitrate Mixtures. See Ethyleneglycol Dinitrate – Nitroisobutyl Dinitrate Mixtures in this vol, p E278-R

Glycol Dinitrate Polymers. See Ethyleneglycol Dinitrate Polymers in this vol, p E278-R

Glycol Dinitrophenylether Nitrate. See under Ethyleneglycolphenylether and Derivatives in this vol, p E279-R

Glycoldiphenylether. See Ethyleneglycol Diphenylether described ad Bis(phenyl)-ethyleneglycol Ether in Vol **2**, p B153-L

Glycol-di-trinitrobutyrate (GTNB). See Ethyleneglycol-di-trinitrobutyrate (EGTNB) in this vol, p E255-R

Glycol Esters and Ethers. See under ETHYLENEGLYCOL AND DERIVATIVES in this vol, pp E247ff

Glycolformal. Same as Glycolmethylene Ether

Glycolglycolate Dinitrate. See Ethyleneglycol-glycolate Dinitrate in this vol, p E256-R

Glycolylguanidine. Same as Glycocyamidine

Glycol, Higher. Under the term "higher glycols" are known glycols contg four or more carbon atoms, such as 2,3-dimethyl-2,3-butanediol (pinacol), used as an intermediate in the manuf of synthetic rubber. Among other higher glycols may be mentioned some which were manufd, although in small commercial quantities, such as 2-methyl-2,4-pentanediol and 2-ethyl-1,3-hexanediol
Ref: S.M. Livengood, "Higher Glycols", pp 284–99 with 137 refs in Curme & Johnston (1952)

Glycolic Acid and Derivatives
Glycolic Acid (Hydroxyacetic or Hydroxyethanoic Acid), $HO.CH_2.COOH$; mw 76.08; colorless, deliquescent crysts; mp 78–79°, bp – decomp; sol in w, alc & eth. Can be prepd by oxidation of glycol or by boiling chloroacetic acid with w or aqueous alkali (Ref 3). Its toxicity is discussed in Ref 2. Used as pesticide and plasticizer, its salts are used as catalysts (Ref 3)
Refs: 1) Beil **3**, 288, (88), [167] & {367} 2) Curme & Johnston (1952), 317 3) Cond-ChemDict (1961), 543-R

Glycolic Acid Azide, Glycolyl Azide, Hydroxy Acetazide or β-Hydroxyethanoylazide (Glykolsäureazid or Oxyessigsäureazid, in Ger), $HO.CH_2.CO.N_3$; mw 101.07, N 41.58%, OB to CO_2 –55.4%, OB to CO –23.7%; bipyramid crysts, mp 189°; sol in eth. Can be prepd by treating hydroxyacethydrazide in cooled HCl with aq Na nitrite. Explodes on heating (Refs 1 & 2)
Its ethyl, propyl and isoamyl esters are also expl compds (Ref 1)
Refs: 1) Beil **3**, 244, (94) & [175] 2) T. Curtius et al, JPrCh [2] **52**, 225(1895) & JCS **70**, 35(1896) 3) L. Jones & D. Powers, JACS **46**, 2520(1924)

Glycolic Acid, Methylester and Derivatives
Glycolic Acid, Methylester, $HO.CH_2.CO.O.CH_3$; mw 90.07; liq, sp gr 1.1677 at 18°, bp 151.2°, 63°(21mm Hg). Prepd by enclosed heating of sodium glycolate, methanol and chloroacetic acid methyl ester at 160°
Refs: 1) Beil **3**, 236, (91) & [171] 2) L. Schreiner, Ann **197**, 5 (1879)

Glycolic acid, Methylester Nitrate, $O_2N.O.CH_2.CO.O.CH_3$; mw 135.06, N 10.37%, OB to CO_2 –41.4%; colorless liq, bp 165°(dec), 82.5°(28mm Hg), n_D 1.4221 at 15°; sol in alc, benz & eth; insol in w. Prepd from the methyl ester and mixed acid, keeping the temp from rising much
Refs: 1) Beil **3**, 236 2) H. Duval, CR **137**, 1263(1903) 3) A.M. Pujo & J. Boileau, MP **37**, 35–48(1955) & CA **51**, 11244(1957)

Glycolide, 2,5-Dioxo-1,4-dioxane or **Diglycolide**, $H_2C.CO_2CH_2CO.O$; mw 116.06, lflts (from alc), mp 86–7°; sol in hot w; sl sol in alc, acet & chlf; insol in eth. Can be prepd by vacuum distn of Na bromoacetate or of glycolic acid
Refs: 1) Beil **19**, 153, (679) & [175]
2) C.A. Bischoff & P. Walden, Ber **26**, 263(1893)
3) CondChemDict (1961), not listed

Glycolmethyleneether or **Glycolformal.** See 1,3-Dioxalane in Vol **5** of Encycl, pp D1405-R & D1406-L

Glycol, Monoethers of. Several monoethers of glycol, including isobutyleneglycol-2-monoethylether, bp 148° and isobutyleneglycol-2-monomethylether, bp 142° were prepd and patented by Edlund. They were claimed to be suitable as solvents for cellulose esters, gums, resins etc
Ref: K.R. Edlund, USP 1968032 & CA **28**, 5832(1934)

Glycol, Monophenylether of. See Ethyleneglycol Phenylether, also known as Phenyl "Cellosolve" in this vol, p E279-L

Glycolonitrile and Derivatives
Glycolonitrile or Glycolic Nitrile (Formaldehyde Cyanohydrin), $HO.CH_2.CN$; mw 57.05, colorless oil, sp gr 1.104 at 19°, fr p <–72°, bp 183° at 759mm (sl dec); v sol in w, alc & eth, insol in benz. Can be prepd by treating formaldehyde with hydrocyanic acid. Used as solvent and org intermediate (Ref 3)

Following description about its expln is given in Ref 4: Less than 2 weeks after a double distn from ethanol, glycolonitrile stored in a screw cap bottle polymerized, generating enough heat and pressure to shatter the bottle
Refs: 1) Beil **3**, 242, (93) & [174] 2) A.J. Ultée, Rec **28**, 249(1908) 3) CondChemDict (1961), 543-R 4) Anon, C&EN **44**, No 49, p 50 (28 Nov 1966)

Glycolonitrile Nitrate, $(O_2NO).CH_2.CN$, mw 102.05, N 27.45%, OB to CO_2 –31.3%; oil, bp 69–70°(below 13mm Hg)(dec); sol in w; expl by rapid heating. Prepd by dry vacuum distn of silver nitrate and iodoacetonitrile
Refs: 1) Beil **3**, 243 2) R. Scholl & W. Steinkopf, Ber **39**, 4396(1906) 3) A.F. Ferris et al, JACS **75**, 4078(1953) & CA **49**, 8158(1955)

Glycolphenylether, Phenyl "Cellosolve" or **β-Phenoxyethanol.** See Ethyleneglycol Phenylether in this vol, p E279-L

Glycol Polymers or **Polyglycols.** See Ethyleneglycol Polymers, Nitrated in this vol, p E279-R

Glycol, Tertiary Ethers of. Several tertiary ethers of glycol, including: tert-butylmonomethylether of glycol, bp 131–32°; tert-butylmonoethylether of glycol, bp 147.5°; and tert-butylmonobutylether of glycol, bp 83.4° at 20mm were prepd by Evans & Edlund who proposed them in the expl industry as solvents and gelatinizers for cellulose esters, resins etc
Ref: T. Evans & K.R. Edlund, USP 1968033 & CA **28**, 5832(1934)

Glycol Trinitrophenylether Nitrate. See under Ethyleneglycol Phenylether and Derivatives in this vol, p E279-R

Glycolurethane and Derivatives
Glycolurethane or Ethyleneglycolurethane, $H_2N.CO.O.CH_2.CH_2OH$; mw 105.08; liq, sp gr 1.285 at 20°, bp 235°, 130–2°(1mm Hg), mp 30°(Ref 4), n_D 1.4652 at 20°; sol in w & alc. Prepd by reacting ammonia with glycol carbonate, $O.CH_2.CH_2.O.C(O)$ at ambient temp
Refs: 1) Beil, not found 2) J.D. Malkemus, USP 2627524(1953) & CA **48**, 712(1954)
3) G. Desseigne, MP **38**, 435–39(1956)
4) R. Delaby et al, Bull(Fr) **1956**, 214 & CA **50**, 9382(1956)

N-Nitroglycolurethane Nitrate,

$O.CH_2.CH_2.ONO_2$; mw 195.05, N 21.54%, OB to CO_2 –12.3%; wh crysts, mp 78.6°; sl sol in cold w, benz & chlf; sol in hot w; v sol in alc, eth, acet & et acet. Can be prepd in 79% yield by adding glycolurethane (1 mole) to 8 mols 98% nitric acid, with stirring at temp not exceeding 10°. Can be detonated by shock and is flammable. Used as gelatinizer for NC and as component of liquid & solid expl compns

Refs: 1) Beil, not found 2) G. Desseigne, FrP 1094959 (1955) & CA **53**, 1159 (1959) 3) G. Desseigne, MP **43**, 109–26 (1961) & CA **58**, 6691 (1963)

Glycoluril or **Glyoxaldiureide,** called in CA **Tetrahydro-imidazo[4,5-d] imidazole-2,5(1H,3H)-dione.** See ACETYLENEDIUREIN AND DERIVATIVES in Vol 1, p A65

Dinitroglycoluril. One isomer, 1,3-, prepd by A.P.N. Franchimont & E.A. Klobbie, Rec **7**, 18–19 & 246 (1888) and Rec **8**, 290–91 (1889) is briefly described in Vol 1, p A65-R, while other isomer: *Isodinitroglycoluril,* also known as x, x′-Dinitroglycoluril is listed on p A66-L

Although no later refs are found in CA thru 1971 for mono thru tetranitro derivs, the dinitrocompd was prepd for NDRC by R.L. Shriner and described in OSRD Rept **2054** (1943), pp 11–12. He nitrated glycoluril with concd nitric acid in presence of acetic anhydride at a temp of 5°. The yield was theoretical. He assigned to it the formula of 1,4-Dinitroglycoluril,

$O_2N.NC(O)NH.\overline{CH.N(NO_2)C(O)NH.CH}$. The compd darkened at 305° without melting and burned quite rapidly, leaving little ash. It was sol in acet, sl sol in w or alc, insol in benz or chlf. Its Ballistic Power was 101% TNT, sensitivity near that of PETN and vacuum stability at 100° unsatisfactory. Was considered by Shriner as unsuitable for military purposes

Three dinitro isomers: 1,3-; 1,4-; and 1,6- were prepd in 1952 by I. Minsky et al at PicArsn and described in Memorandum Report **PAMP 16** (1952)

Nitration of glycoluril with 100% nitric acid by the method similar to that of Franchimont & Klobbie yielded (84% of theory) a mixt of 1,3-, 1,4-, and 1,6-isomers. The 1,3 isomer (10% of total) was unstable and could be decompd by boiling water. The remaining mixt of 1,4- and 1,6-isomers was washed with ethanol and dried

Procedure for Nitration. Add with stirring 10g of glycoluril (predried at 70°) to 100ml of absol nitric acid at RT. Warm the soln to 55° (in a water bath), while stirring for 1 hour, and pour slowly into 200ml of boiling w. The resulting evoln of gas is caused by decompn of 1,3-isomer. Cool the slurry in an ice-bath to 0°, filter off the ppt, wash it with ice-cold w until neutral to litmus, followed by rinsing with ethanol and drying

The resulting mixture of **1,4-** and **1,6-Dinitroglycolurils** had mw 232.12, N 36.21%, OB to CO_2 –27.6%, OB to CO 0%, mp – deflgr betw 270 & 280° (vs 475° for TNT). Franchimont & Klobbie gave 217° as deflgm temp (probably for impure compd)

Brisance by Sand Test1 42.1g sand crushed, vs 48g for TNT (minimum detonating charge of Tetryl was 0.089g)

Heat of Combustion at C_v. 1901cal/g

Heat Test at 100°. Loss in wt 0.41% 1st 48 hrs, 0.0% 2nd 48 hrs and no expln

Impact Sensitivity by BurMinesApp. 2kg wt – 50cm, vs 60–100 for TNT and by PicArsnApp 7 inches, vs 14 inches for TNT

ISP (Calculated). Specific Impulse of gases 197 lbs, sec/lb, vs 215 for Guncotton (13.15%N)

Vacuum Stability Test at 90°. 0.25cc gas evolved by 1g in 40 hrs, vs 0.10cc for TNT

Tetranitroglycoluril or **Tetranitroacetylenediurein.** The compd was prepd and investigated in 1952 at Picatinny Arsenal by I. Minsky et al and described in Memorandum Report **PAMA 16** (1952):

1,3,4,6-Tetranitroglycoluril,

$O_2N.N.C(O)N(NO_2)\overline{CH.N(NO_2)C(O)N(NO_2)CH}$; mw 322.06, N 34.78%, OB to CO_2 +5.0%; hygroscopic crysts, mp – decomp ca 225°; sol in NMe, decompd by w & hot ethanol; insol in glac AcOH, eth, dioxane, toluene or xylene.

Can be prepd by nitration with $HNO_3 + P_2O_5$ of either glycoluril or its dinitro compd
Procedure: Add quickly with stirring 10g of glycoluril (predried at 70°) to a mixt (preheated to 50°) of 150ml absol HNO_3 and 15g P_2O_5, contd in a 3-neck 300-ml round bottom flask, provided with thermometer, stirrer and reflux condenser. After stirring for 1.5 hours at 50°, cool the flask with contents in an ice bath to about 0° and pour the contents slowly on about 250g of crushed ice. Leave standing overnight in a cold place, then filter and wash the ppt with ice-cold w until neutral. Finally wash with ice-cold ethanol, then with ice-cold ether and dry the crysts in air. The usual yield was 30% of theory
Note: When nitrating dinitro- to tetranitrocompd, reduce the amt of HNO_3 to 100ml, the amt of P_2O_5 to 14g and the time of stirring at 50° to 1 hour

Tetranitroglycoluril is an expl more brisant than TNT, but comparable to PETN. It is extremely sensitive to impact and of inadequate thermal stability

The following props were detd at PicArsn:
Brisance by Sand Test. 61.9g sand crushed (vs 48.0 for TNT & 62.7 for PETN)
Explosion Temperature. 133°(vs 225° for PETN)
Heat Test at 75°. exploded in 30 minutes
Impact Sensitivity. 2kg wt, PicArsnApp, 2 inches (PETN 6 inches)
Impact Senditivity, 2kg wt, BurMinesApp, 6cm (PETN 17cm)
ISP (Specific Impulse of gases), calcd 195 lbs, sec/lb, vs 215 for NC (13.15% N)

Glycolyl. The monovalent radical $HO.CH_2.CO-$ or divalent radical $=C_2H_2O$

Glycolyl Azide. See Glycilic Acid Azide under Glycolic Acid and Derivatives in this vol

Glycolylurea and Derivatives. See under Hydantoin and Derivatives in Vol 7 of Encycl

Glycoside. See Glucoside

Glykol. Ger for Glycol

Glyoxal or **Ethanedial,** O:CH.CH:O; mw 58.04, yel crysts, sp gr 1.14 at 20°(1.26 at 20/20°), mp 15°, bp 51° at 776mm, n_D 1.3826 at 20°. Its vapor is green and burns with a violet flame; sol in w, alc & eth. Was first prepd in 1881 (Refs 1 & 2) by oxidizing acetaldehyde with nitric acid. This and other methods are described by Curme & Johnston (Ref 4, pp 125–26)

It polymerizes on standing or in presence of trace of w, forming "trimeric glyoxal"

Its toxicity is discussed by Curme & Johnston (Ref 4, p 317). In skin absorption it is about 1/10th as toxic as formaldehyde

The industrial utilization of glyoxal is largely based on the advantage which may be taken of its two aldehyde groups, its lack of volatility from aqueous solns, and its inoffensive color. Among its uses may be cited insolubilizing of proteins, polyvinyl, alcohol, starch, etc (Ref 4, p 127) (See also Ref 5). The use of its commercial aqueous solns was reviewed by Bohmfalk (Ref 3)
Refs: 1) Beil **1**, 759, (393) & [815] 2) N. Liubavin, JRussPhysChemSoc **1881**(1), pp 329 & 495 and Ber **14**, 1713 & 2685(1885) 3) J.F. Bohmfalk et al, IEC **43**, 786(1951) 4) Curme & Johnston (1952), 125–28 & 317 5) CondChemDict (1961), 545-L

Glyoxaldioxime. See Glyoxime in this Vol

Glyoxaline (or Imidazole) and Derivatives. See 1,3-Diazole and Derivatives in Vol 5, pp D1165-R & D1166-L

Glyoxiline or **Glyoxylene.** A Dynamite patented in 1867 by F. Abel: NG 65.5, NC 30.0, K nitrate 3.5 & Na carbonate 1.0% (Ref 1). Accdg to Naoúm (Ref 3), Abel saturated a mixt of K nitrate and Guncotton and called the product "Glyoxilin". Accdg to Daniel (Ref 2), Broderson patented in 1867 in England a similar compn, but it never was used in practice

Refs: 1) Daniel (1902), p 1 (Abel)
2) Daniel (1902), p 355 (Glyoxiline)
3) Naoúm, NG (1928), 282

Glyoxime and Derivatives
Glyoxime, Glyoxaldioxime or *Oximinoacetaldoxime*, HON:CHCH:NOH; mw 88.05, N 31.82%, OB to CO_2 –72.7%, colorless plates (from w), mp 178° (dec at 172°); sol in hot w, alc & eth. Can be prepd from glyoxal (1 mol) and hydroxylamine hydrochloride (2 mol)
Refs: 1) Beil **1**, 761–2, (394) & [818]
2) M. Wittenberg & V. Meyer, Ber **16**, 505 (1883)

Nitroglyoxime (Nitro-oximino-acetaldoxim, in Ger), HON:CH.C(NO_2):NOH; mw 133.03, N 31.58%, OB to CO_2 –18.0%; ndls (from w or other solvents), mp ca 111° (dec), bp – expl on heating above mp; sol in w, decompg in hot and imparting to the soln a strong acid reaction; sol in alc, eth & acet; v sl sol in cold benz, more in boiling benz; insol in chlf & petr eth. Can be prepd by treating an ethereal soln of glyoxime with fuming nitric contg NO_2 or by treating glyoxime with NO_2 alone

It is an expl compd. Being acidic, it forms salts on being treated with bases. The following are expl:
Hydrazine Salt, $C_2H_3N_3O_4.N_2H_4$; brick-red pdr, mp 95° (dec), puffs off at higher temp; sol in w & hot alc; sl sol in cold alc; insol in eth & chlf; dissolves in acet with decompn (Ref 2, p 2750)
Lead Salt (Basic), $(C_2H_2N_3O_4)Pb.O.Pb(C_2H_2N_3O_4)$, yel solid, mp – expl on heating. Was prepd by adding an aqueous soln of Pb acetate to aq soln of Nitroglyoxime (Ref 2, p 2750)
Potassium Salt, $C_2H_2N_3O_4.K$, brownish-yel ndls (from abs alc), mp (explodes on heating) (Ref 2, p 2748)
Silver Salt, $C_2H_2N_3O_4.Ag+H_2O$, red ndls, mp (deflgrates on heating) (Ref 2, p 2748)
Refs: 1) Beil **3**, (217) 2) E. Bamberger & U. Suzuki, Ber **45**, 2740–58 (1912) 3) C. Upiani, Bazz **42**, I, 255 (1912)

GM–1. Ger for liquid nitrous oxide used during WWII as fuel booster for airplane engines
Ref: Maj R.E. Richardson et al, CIOS Rept **25–18** (1945), p 5

Gmelin, L. (1788–1853). German chemist noted as the author of now known as "Gmelin's Handbook der anorganischen Chemie"
Refs: 1) Hackh's Dict (1944), 385-L
2) L.M. Miall & D.W.A. Sharp, eds, "A New Dictionary of Chemistry", J. Wiley, NY (1968), 270–1

Gnom. A pocket size magneto-electrical exploder manufd by Schmitt & Co at Küppertesg (Germany) during the 2nd half of 19th century. Its illustrated description is given in Ref
Ref: Daniel (1902), 243–44

"Gob" Fires in Coal Mines. Under this name are known spontaneous combustions in coal mines arising in waste (gob) piles. When the area had been partially or completely sealed off, the heat could distil combustible gases from residual coal in the vicinity and create an expl mixt if the available oxygen had not been depleted at too fast a rate. An odor known as "gob stink" sometimes served as a warning to workers of the existence of one of these fires
Refs: 1) T.N. Mason & F.V. Tideswell. "Safety in Mines Research Board", GtBritain, Paper No **75** (1933) & CA **27**, 2813 (1933)
2) Ibid, Colliery Guardian **149**, 97–100 (1934) & CA **28**, 6312 (1934) 3) W. Whitmaker; Science Progress **29**, 438–55 (1935) & CA **29**, 2743 (1935) 4) T.N. Mason & F.V. Tideswell, TransInstMiningEngrs(London) **98**, 1–20 (1939) & CA **34**, 5664 (1940) (The detection of "gob" fires)

Goddard, Robert H. (1882–1945). American scientist, who is considered in the USA as the father of modern rockets. He worked mainly on the mechanical aspects of rockets but also made important developments in rocket fuels

Refs: 1) Willy Ley, "Rockets, Missiles and Space Travel", Viking, NY (1957), pp 107–08 2) Rocket Encycl (1959), pp 191–95 (More detailed description of Dr Goddard's education, contributions, and more than 200 patents in rocket engineering)

GOER Vehicles are all terrain, floatable wheeled vehicles consisting of a front power unit and a trailing functional unit – 8 ton cargo, 2500 gal tanker, or 10 ton wrecker. These were given some field experience in the Vietnamese arena, and are undergoing further testing. The normal suspension system has been dispensed with, the function absorbed by the 10-ply 18.00 x 33 tires which also serve to give the necessary ground clearance. Manufacturer is Caterpillar Tractor Co
Ref: J.B. Hayes, NatlDefense (formerly Ordnance), **58**, 115, Sept-Oct 1973

Goetz Powder. K chlorate 34.5, glucose 34.5, Pb picrate 10.3, charcoal 10.3, sulfur 6.9 & amorphous phosphorux 3.5%
Refs: 1) Daniel (1902), 355 2) Giua, Trattato **6**(1)(1959), 400

Golovine Explosive. Amer primary expl listed by Giua, Trattato **6**(1)(1959), p 398 as of 1905. Its compn is given in Vol **3** of Encycl, p C392-L, under Coke

Gomez Powders. Mixtures of K chlorate and nitrate with Pb nitrate and acetate
Refs: 1) Daniel (1902), 356 2) Giua, Trattato **6**(1)(1959), 396

Gomme (Fr for Gum). In the Fr and Belg expls industry, it is a general term for gelatin expls (gélatines explosibles) consisting of NG gelatinized by NC, to which an alkali or Amm nitrate and carbonaceous material may be added
Following is the list of such expls:
Gomme A: NG 92 & NC 8% (Corresponds to Amer Blasting Gelatin)
Gomme B: NG 83, NC 5, K (or Na) nitrate 10 & sawdust 2%
Gomme B AM: NG 60, NC 3, Amm nitrate 31 & cellulose 6%
Gomme C: NG 74, NC 6, K (or Na) nitrate 15.5 & sawdust 4.5%
Gomme D: NG 69.5, NC 5.5, K nitrate 24.75 & Na carbonate 0.25%
Gomme D de Cugny or Gomme E: NG 49, NC 2, Na nitrate 36, sawdust 10 & flour 3%
Gomme J or Gomme à 92: Same as Gomme A
Gomme M: NG 74, NC 6, K nitrate 15.5, flour or woodmeal 4 & Mg oxide 0.5%
Gomme MB: NG 70, NC 4, K nitrate 16, flour or woodmeal 9,5 & Mg oxide 0.5%
Gomme N: NG 25, NC 1, K nitrate 10 & Amm nitrate 64%
Gomme NB: NG 25, NC 1, Na nitrate 10 & Amm nitrate 64%
Gomme au nitrate d'ammoniaque: NG 37.5, NC 2.5, Amm nitrate 55 & cereal flour 5%
Gomme incongélable de la Nobel française: NG 75, NC 5, K (or Na) nitrate 10, DNT 8 & sawdust 2%
Gomme incongélable de la Société d'Explosifs et de Produits chimiques: NG 64.4, NGc 16.6, NC 5, K (or Na) nitrate 10 & sawdust 2%
Gomme supérieure or Gomme extraforte. Same as Gomme A
Gomme de sûreté de Cugny; Gomme NP: Same as Gomme N (above) and
Gomme NS – same as Gomme NB (above)
Refs: 1) L. Lheure, MP **11**, 222–24 (1901) 2) Daniel (1902), 328–29 3) Gody (1907), 365, 376 & 377 4) Naoúm, NG (1928), 310 & 404 5) Pepin Lehalleur (1935), 334–36

Gonflement, Coefficient de. Fr for Swelling Coefficient. See Coefficient de gonflement in Vol **3**, p C389-R

Gonne (Cannon). The earliest weapon using BlkPdr, including cannon, hand cannon, pot de fer & totenorgel, dates from around 1300. Gonnes were slow to evolve. The monsters lacked mobility, the little bombards or muskets or arquebuses shot a weak chge. Both took a long time to fire. In time, the gonne did improve, but their military application took many years longer. Although cannons are recorded as being used in land action at Seville in 1247,

it was a century later (1351) in naval action between the Moors & Spanish that ship cannons won the battle. The Moors are recorded as using guns against Christians of the Alcazar in 1343. Venetians are supposed to have employed guns against the Genovese in 1380

See also **CANNON** in Vol **2** of Encycl, pp C26-Lff

Refs: 1) M. Lindsay, "One Hundred Great Guns: An Illustrated History of Firearms", Walker & Co, NY (1967), pp 29–33 2) Staff, Ordn **53**, 99 (1968) (A review of the above book)

Gooch, Frank Austin (1852–1929). An American chemist, noted for analytical methods, especially as the inventor of "Gooch Crucible". It is a crucible with perforated bottom packed with glass wool or asbestos and placed in a funnel inserted in the stopper of vacuum flask. Can be used for filtering corrosive liquids

Ref: Hackh's Dict (1944), 386-L

Goodyear Powder. Black Powder, patented in England in 1865, contd saltpeter, sulfur, charcoal, and a small quantity of caoutchouc or guttapercha

Ref: Daniel (1902(, 356

"Goop" is the name given to the crude intermediate product obtd in the manuf of magnesium at the Henry Kaiser plant. It consists of a mixt of ultrafine particles of Mg, Mg carbide, magnesia, carbon, naphtha & asphalt, all in the form of thick paste. The material proved to be a suitable source of Mg in prepn of incendiary compns for bombs. The most successful of such mixtures was known during WWII as **Pyrogel** or **PT-1**

Refs: 1) R.W.Hufferd, ChemMetalEngrg **53**(10), 110–13(1946) 2) A.C. Byrns, ChemEngrg-Progress **43**, TransAmerInstChemEngrs, 172–73(1947) & CA **41**, 3412(1947)

Goriunov, P.M. (1901–1943). Russian engineer who designed some automatic weapons, such as machine gun **SG-43** (Stankovaya Goriunova-43)

Ref: W.H.H. Smith & J.E. Smith, "Small Arms of the World", Stackpole, Harrisburg, Pennsylvania (1960), p 159

Gossot, Fernand (1853–1935). French general specializing in ballistics. Numerous publications in Mém de l'Artil de la Marine, Mém de l'Artil Française, Révue de l'Artil, and Mém des Poudres. In collaboration with R. Liouville compiled "Balistique Intérieure", Baillière, Paris (1922) and new edition published in 1933 by Gauthier-Villars, Paris

Ref: Anon, MAF **15**, after page 646, pp I to VI (1936)

Gotham Explosive (Amer) consisted of NG 66, K chlorate 20, K nitrate 4 & pulverized cinchona or oak-tree bark 10%

Refs: 1) Daniel(1902), 356 2) Giua, Trattato **6**(1)(1959), pp 344 & 400

Goudron. Fr for tar or pitch

Note: Accdg to "New Century Dictionary" (1948), Appleton Century Crofts, NY, the name "goudron" was given to a bundle of sticks soaked in pitch, oil or the like and used either for lighting or setting fire to enemy's work

Government Book Stores

The US Govt sells publications thru three outlets. All purchases must be prepaid. While GPO deals in general publications, occasional titles will be of interest to the explosives and pyrotechnics industry

GPO – Superintendent of Documents
US Govt Printing Office
Washington, DC 20402

DDC – Defense Documentation Center
Cameron Station
Alexandria, Va 22314

NTIS – National Technical Information Service
US Dept of Commerce
Springfield, Va 22151

The two technical houses are DDC and NTIS. DDC used to be ASTIA. It handles classified or otherwise restricted documents and you must be registered there. This is important. If you are not registered, write for information but don't waste time asking for documents. (Limited documents – numbers followed by the letter L – require additional approval by the issuing agency). NTIS used to be Clearinghouse.

It sells open material. Documents in both collections sre indexed, the indexes being available in most technical libraries. Most documents from DDC and NTIS cost $3 for hard copy and 95¢ for microfishe, while GPO publications are priced individually
Ref: G. Cohn, Expls&Pyrots **5**(12), 1972

GPF (Abbrn for Grand Puissance Filoux). French heavy artillery weapon (such as 155-mm cannon), successfully used during WWI

"G" Pulver. (Flashless Propellant of Gallwitz) (Ger). See Vol **3**, pp C511-R & C512-L, Vol **5**, p D1536-R to D1537-R and in PATR **2510** (1958), pp Ger 70-R & Ger 71-L

Gräbe (or Graebe), Carl (1841–1927). A German org chemist who established the constitution of naphthalene, anthracene and phthalic acid, etc. Co-synthesized with Carl Liebermann (1842–1914) alizarine
Ref: Hackh's Dict (1944), 386-R (Graebe); 490-L (Liebermann)

Grade Construction. The use of explosives for grading work can be divided into the following classifications: 1) clearing and grubbing; 2) ditching and drainage; 3) excavation; 4) fill settlement; 5) production of surfacing material and ballast. As a result of rapid development of drilling equipment (percussion and rotary type machine) borehole diameters of 2½ to 5 inches are used, but larger holes are possible. Types of explosives applicable to highway blasting include Forty per cent Dupont Gelatin, Gelex 1 or 2 and Red Cross Extra. Nitramon and Nitramite are used for large holes on highway work. For small dry work Nitramite FR is used considerably, also Nilite and Ammonium Nitrate prills and oil can be used under similar conditions if the holes are large enough. Open face shooting requires less explosives, the factor depending largely on the type of rock. Factors usually run 0.50 to 0.75 pound per cubic yard. The result of use of MS delays in grade construction is improved fragmentation and decreased vibration. In addition, they aid in controlling the direction and amount of throw of the broken rock. Primacord and MS connectors are used instead of MS delays for greater safety when spacings between holes are large enough to minimize the chance of cut-offs. For firing, two sizes of condenser discharge type of blasting machines known as CD-32-1 and CD-48-1 with capacities up to 480 and 1200 blasting caps, respectively
Ref: Blasters' Hdbk (1963), pp 413–23 and 15th Edition (1969), 385–93

Grageroff, I.A., Dr (1880–1962). Russian-American chemist, an authority in many branches of explosives. Worked for some time at Apache Powder Co, Benson, Arizona and then during WWII as the Director of Operation of one of the largest TNT Plants in the US located at Meadville, Pennsylvania. Introduced a method of manuf of TNT which speeded up the production threefold and resulted in considerable economy
Ref: B.T. Fedoroff, who worked before and during WWII under Dr Grageroff

Graham, Thomas (1805–1869). A Scottish chemist, pioneer in the study of colloids, the diffusion of gases and dialysis. Developed so-called *Graham Law* which states that velocities of diffusion of any two gases are inversely proportional to the square roots of their densities
Ref: Hackh's Dict (1944), 386-R

Graham Explosive. It was prepd and used in a moist condition in the form of a paste consisting of K chlorate 51.8, sugar 25.9, $K_4Fe(CN)_6$ 20.7 & Pb_3O_4 1.6%
Refs: 1) Daniel (1902), 356 2) Giua, Trattato **6**(1), 1959, p 400

Grain. A unit of the apothecaries, avoirdupois and troy weights (originally that of an average wheat grain), approx equal to 0.0648 gram (Ref 1). In Ordnance, a single piece of a solid expl or solid proplnt, regardless of size or shape, used in a gun or a rocket (Ref 2) (See also Grains of Smokeless Propellants)

Refs: 1) Hackh's Dict (1944), 387-L 2) Glossary of Ordn (1959), 137-L

Grain Dust Explosions. See under Dust Explosions in Vol **5** of Encycl, p D1578-L

Graining is the process of forming material into smaller particles than the original material. One of such processes, *prilling* or *spray graining*, used in manuf of AN, is described in Vol **1** of Encycl, p A314-R. Very often graining can be accomplished by *crushing and grinding* as described in Vol **3** of Encycl, pp C564-R to C567-L (Compare with Grist and with Granulation)

Graining of Smokeless Propellants (Grenage, moulage, étirage ou filage des poudres colloidales, in Fr). This operation is one of the steps in the manuf of smokeless propellants. It is briefly described in Vol **3** of Encycl, under "Colloiding Agents and Colloidal Propellants", p C399-R for single-base propellants and p C400-L & R for double-base proplnts [See also Davis (1943), 302–04]

French method is described by Pepin Lehalleur (1935), 298–99, under the title "Moulage ou étirage"

Grains of Smokeless Propellants. Smokeless proplnts are forms of Nitrocellulose expls with various organic and inorganic additives and are used as proplnts. They are divided by compns into classes of which the single-base and double-base are the most common. They are manufd in quantity in a variety of shapes (Fig) (Ref 2). Cylindrical grains are made in various diameters and lengths depending on the size of the gun. Perforations shown in Fig are for controlling the rate of gas liberation as well as burning time. At constant pressure, the time of burning is proportional to the amount of exposed powder surface, therefore the proplnt is made into accurately sized grains of selected shapes (Refs 1 & 3)

Proplnt grains in the shape of ball, strip, cord and pellet are of *degressive granulation* because their surface area decreases during burning (Ref 2, p 91-L). Single-perforated proplnt grains are of *neutral granulation* because their surface area remains constant during burning (Ref 2, p 194-L). Multiperforated and rosette proplnt grains are of *progressive granulation* because their surface area increases during burning (Ref 2, p 223-R)

Refs: 1) A.J. Phillips, "Smokeless Powder Grains", ArmyOrdn **11**, 195–98 (1930) (Causes and Characteristics of Structural Variations) 2) Glossary of Ordn (1959), 91-R, 194-L &

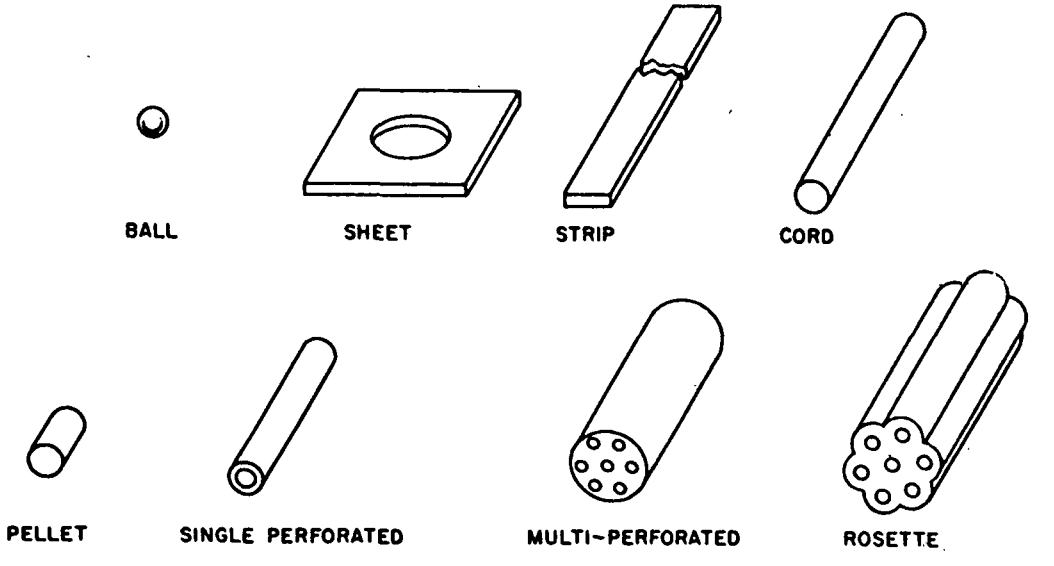


FIG. Typical Shapes of Powder Grains

223-R 3) Anon, "Element of Armament Engineering", Part 1, "Sources of Energy", Engineering Design Handbook, US Army Ordnance Corps Pamphlet **ORDP 20-106** (1960), pp 3-15 to 3-17

Grakrult. Smokeless pdrs patented by Skoglund of Sweden prior to 1889. They consisted of NC, Picric Acid and a radical of carbonic, oxalic or carbamic acids in combination with ammonia or other volatile radical base
Refs: 1) J.W. Skoglund, JSCI **8**, 478(1889) 2) E.C. Worden, "Technology of Cellulose Esters", Vol **1**(1921), Pt 3, p 1637

Granada, Fábrica de Pólvoras y Explosivos de. One of the best known Spanish Plants manufg Explosives and Propellants. It was founded in 1442 while nearly all Southern Spain, including Granada and its fortress Alhambra was in the hands of Arabs. When Granada was reconquered by Spain in 1492, the plant became the property of Royal Spanish Government. The plant was greatly extended in 1908. Its detailed description is given in Refs 1 & 2 and a briefer description is in Ref 3

The following Ordnance plants were constructed in Spain in later years: Fábricas de Artilleria de Sevilla in 1565, – de Toledo in 1780, – de Oviedo in 1794, – de Trubia in 1795 and de Murcia some time during the rein of Felipe II (1527–1598); La Pirotecnia de Sevilla in 1846. These Govt plants and private plants, such as the Sociedad Anónima de Dinamita de Galdácano (Bilbao) manufd high quality expls, proplnts, ammunition and weapons, most of them for export to Mexico, Central & South America and Cuba
Refs: 1) Col R. Aranaz et Izaguirre, Memorial de Artilleria, May 1908 2) Ibid, SS **3**, 248, 283 & 306 (1908) 3) Vivas, Feigenspan & Ladreda, Vol **3**(1948), p 79 (Footnote) and pp 79–93

Granata. Russ for Grenade, whereas *Snariad* is for Artillery Projectile (Shell)

Granate. Ger for Grenade and for Shell

Granadfüllung. Ger for Bursting Charge for Shells

Granatfullung 88 or Grf 88. Ger name for Picric Acid (PA), which started to be used for Ger Shells in 1888

Granatfüllung 02 or Fp 02. Ger name for TNT (Trotyl), which started to be used as filler for Ger Shells in 1902

Granatprobe. Ger for Fragmentation Test

Grandcamp Explosion. An explosion of 2300 tons of Ammonium Nitrate (AN) fertilizer, which took place aboard the SS Grandcamp on April 16, 1947, while the ship was being loaded in the harbor at Texas City, Texas. The explosion was started by a fire in one of the holds

As a result of the explosion, a fire developed in one of the holds of the SS High Flier (moored 600 ft away from the Grandcamp), contg sulfur. This fire spread to one of the holds contg 960 tons of AN which exploded on April 17

These explosions caused a loss of life of 600 persons and property damage of about $50 million
Ref: PATR **2700**, Vol **1**(1960), pp A359-L to A361-R and Refs 54 to 64, listed on p A363-L

Granodizing Process, developed by the American Chemical Paint Co, Waterbury, Conn for rust prevention of iron, steel, zinc or cadmium consists in immersing the material in a hot soln of acid zinc phosphate. This operation forms an acid resistant coating. The resulting material may be painted

The process is fundamentally the same as "parkerizing"
Refs: 1) D.D. Sager, PA Tech Repts **887**(1938) & A.J. Phillips, **945**(1938) 2) M.Merlut-Sobel, "Metals and Alloys Dictionary", ChemPubgCo, NY (1944), p 99

Granular Explosives of Whetstone. A Dynamite is made by emulsifying a molten HE, such as TNT, by the aid of an emulsifying agent, such as agar-agar, in the melt comprising AN and at least one compd nonvolatile at the temp of the melt and which lowers the mp of the AN such as $Ca(NO_3)_2.4H_2O$, $Pb(NO_3)_2$, alkali-metal nitrate, $Mg(NO_3)_2.6H_2O$, anhyd NaOc, Na benzene sulfonate, sucrose, acetamide, urea or dicyanodiamide. The emulsion freezes at above 46° and is free from uncombined water
Refs: 1) J. Whetstone, BritP 597716(1948) & CA **42**, 4349(1948) 2) J. Whetstone, USP 2460375(1949) & CA **43**, 2776(1949)

Granular Smokeless Propellants. An improved smokeless proplnt is produced by providing a central perforation with or without radial slots in addn to porosity produced by leaching out a water-sol cryst ingredient. Thus, a proplnt compn is made from colloided NC (13.25% N) 100, Pb_2NH 1.1 & KNO_3 45–50 parts with the aid of a volatile solvent, and is extruded thru a die so as to produce the central perforation with or without radial slots
Refs: 1) S. Gordon, BritP 626955(1949) & CA **44**, 3528(1950) 2) S. Gordon, USP 2575871(1951) & CA **46**, 2299(1952)

Granulation of an Explosive or Propellant. The size of the grains can be ascertained by sifting the material thru a series of sieves with different sizes of aperture and weighing the proportions retained on each sieve (Ref 1)

The sieves used in the USA are US Natl-BurStds Sieves. They are listed in Table 1, p A674 of Ref 2 in comparison with Tyler, British and German Sieves
Refs: 1) Marshall **3**(1932), 124 2) Encycl of Expls, PATR **2700**, Vol 1(1960), p A674

Granulation Process. It is an act of forming solid material into particles of desired size and shape, usually of larger size than original powder. It is one of the branches of "size enlargement" and differs in this respect from **graining** which deals usually with "size decreasing". Very often no distinction is made betw the terms "granulation" and "graining"

The methods of producing granules from powdery substances may be subdivided into:
a) *Granulation with a binding agent* consists in moistening the powder with a binding soln, followed by screening, rotary drying etc
b) *Granulation by fusion* consists in agglomerating the material contg binding agents such as pitch, paraffin, wax etc, into sheets, followed by cooling and reducing to granules of the desired shape. Sometimes, material can be fused directly to granules of the desired shape
c) *Granulation by briquetting* consists in agglomerating the powdered material into small size briquettes, also called "slugs", followed by grinding and screeing
d) *Granulation by spray drying* consists of suspending the material in a liquid and spraying the suspension thru a nozzle into a drying chamber by means of hot compressed gas
e) *Granulation by the Cabot spheronizing process* consists of subjecting the material to turbulent mechanical action in mixers with specially designed agitators. The material gradually agglomerates into spherical form with large reduction in bulk density. The process can be usually speeded up by using small spherical "starters" sifted from the previous batch
f) *Granulation by dissolving a solid material in a liquid and running the solution in a thin stream into another liquid* which is nonsolvent for the solid, but is miscible with the first liquid. For instance, TNT or NC may be dissolved in acetone and then run into water
g) *Granulation by melting of a solid material* and running it thru a screen situated on the top of a tower, down which the material flows. This method can be used for the prepn of shot. One of such towers may be seen in Baltimore, Maryland
h) *Granulation by Olsen process*, used in the prepn of BALL POWDER. Process is described in Vol **2** of Encycl, PATR **2700**(1961), pp B11-R to B16-L
Ref: Perry (1950), 1187–88; and (1963), pp 8-60ff

Granulite. A British, large, irregular-grain, smokeless proplnt consisting of NC 46.3, Ba nitrate 45.5, paraffin 7.8 & moisture 0.4%.

Paraffin was used for coating the grains
Ref: Daniel (1902), 357

Granulometry of Explosives. Detn of granularity by sieving is often thought to be unprecise. However, it is possible to make the measurement precise if certain precautions are observed. In particular, the product must spread out smoothly on the screen without flying out, and sieving must be continued until nothing is left. Manual sieving is satisfactory, but faster, more precise results are obtd with a mechanical sieve. The crystals of many expls are electrified by friction during sieving. This seriously affects the results. Addn of 1 or 2% of graphite helps in some cases. For fine powders, accurate results can be obtd only if the sieving is done under water
Ref: P. Aubertein, MP **37**, 139–48 (1955) & CA **50**, 17453 (1956)

Grape-shot or **Case-shot.** Accdg to Greener (Ref 1), most of the early firearms shot arrows, stones, and iron shots, but in Germany the mortars were filled up with small stones about the size of walnuts, the first form of what was afterwards long known as *grape shot*, also called *hail-shot*. Some Ger states forbade the use of these shots

Marshall (Ref 2) states the following: "Formerly *case shot* was used against troops at short range. It consisted of a case containing a large number of bullets, which spread out from the muzzle of the gun, the case being broken up in the bore. The principal case shot were *grape*, *canister* and *shrapnel case*. They are not used much now and their place has been taken by *shrapnel shell* (invented in 1784 by Lt of Brit Army Shrapnel), and *machine guns*

There was also so-called *chain-shot*, said to be invented in Holland by DeWitt, about 1666. It consisted of two balls or half balls united by a chain. It was fired against the rigging of ships

One type of grape-shot consisted of a number of cast-iron balls, about the size of grapes, packed in layers in a cylindrical container, equaling in diam the bore of the gun. A round usually consisted of 9 balls in tiers of three. The breaking of the cylinder discharged the shots in all directions (See also "Canister" in Vol **2** of Encyc, p C244-R)
Refs: 1) W.W. Greener, "The Gun and Its Development", 9th Ed (1910), reproduced by Bonanza Books, NY, p 28 2) Marshall **1** (1917), p 31

Graphic Methods. Pencil and paper methods that employ the geometry of a plane to express mathematical relationships and to carry out mathematical operations in analog form. Some topics under this definition are plotting of experimental data, curve fitting and development of empirical formulas, graphical interpolation and extrapolation, nomographs and alignment charts, graphical differentiation and integration, graphic statics
Refs: 1) W.C. Brinton, "Graphic Methods for Presenting Facts", McGraw-Hill, New York (1914) 2) A. Schultze, "Graphic Algebra", MacMillan Co, New York (1933) 3) J. Reilly & W.N. Rae, "Physico-Chemical Methods", VanNostrand, NY (1943), pp 161–70, 180–84 4) McGraw-Hill Encyclopedia of Science and Technology **6** (1971), pp 286–87

Graphigen Rocket Fuel. See under Graphite-Containing Rocket Fuel

Graphite (Black Lead, Plumbago, Mineral Carbon), C, at wt 12.01; d 2.25, mp 3527°, hardness by Mohs scale 1 to 2. Black, cubic crysts with metallic lustre; when in powdered form it is steel-gray to black in color with a dull metallic lustre. It occurs in nature in many places. The best grades of commercial graphite are from Ceylon. It may also be prepd artifically by heating other forms of carbon in an electric furnace. It is insol in any of the known solvents, but is sol in molten iron. It is not attacked by some strong oxidizing agents with formation of "Graphitic Oxide" (See under). If the oxidation of graphite by nitric acid is conducted in the presence of catalysts, such as vanadic acid or silver nitrate, mellitic acid, $C_{12}O_{12}H_6$, is obtd

Graphite is used extensively in commerce. In the expl industry it is used for glazing grains of BkPdr and smokeless proplnts in order to render them less hygroscopic and

make them electrically conductive, so that they cannot accumulate static charges. Graphite used in the manuf of smokeless proplnt should be quite free from sulfides and acids and should contn only a trace of silicates (Refs 1 & 2)

Graphite has found applicatuon in the prepn of Tetryl pellets. In this case, a small quantity of powdered graphite is mixed with powdered Tetryl and the mass is pelleted. The presence of graphite eases this operation because it makes the Tetryl less sticky (ie graphite acts as a pelletizing lubricant)

Some uses of graphite as an ingredient of expls, proplnts and pyrotechnic compns are listed in Vol **2** of Encycl, under CARBON, pp C54-L to C55-R and in some Refs of pp C55-R to C56-R

Electronic properties of graphite are described in Vol 8 of Ref 6, while its thermal props are in Vol 10. In Vol 11 is deecribed highly oriented pyrolytic graphite

Refs for Graphite: 1) Marshall **2**(1917), 707 2) R.G. Woodbridge, USP 1390740(1922) & CA **16**, 344(1922) 2a) Davis (1943), 291, 293, 295, 306 & 330 3) Karrer (1947), 520 4) Kirk & Othmer, 2nd edit 4(1964), 158ff 5) CondChemDict (1961), 550; (1971), 426-L 5a) AMCP **706-187** (1963), 140–42 6) P.L. Walker, Jr & P.A. Thrower, Edits, "The Chemistry and Physics of Carbon", Marcel Decker, Inc, NY 10016(1973): Vol 8, "The Electronic Properties of Graphite" and "The Behavior of Fission Products Captured in Graphite"; Vol 10: "The Thermal Properties of Graphite" & "Lamellar Reactions in Graphitizable Carbons"; and in Vol 11: "Highly Oriented Pyrolytic Graphite" 7) G. Cohn, Edit, Expls&-Pyrots **7**(1), 1974 (Lists Vols 8–11 of the above book and suggests that the paper on electronic properties in Vol 8 may be of interest in connection with carbon bridge detonators)

Graphite, Colloidal. It may be either natural or artificial graphite ground to 1 micron particle size, coated with a protective colloid, and dispersed in a liq. One selects the liq carrier – water, oil, or synthetics – for the use intended. The two outstanding props of colloidal graphite dispersions are: 1) the particles remain in suspension and 2) the particles "wick", ie are carried by the liq to most places the liq penetrates

Ref: Clark & Hawley (1966), p 495 (Graphite by S.B. Seeley)

Graphite-Containing Rocket Propellants. *Graphigen* rocket fuel is prepd from graphite, K perchlorate and 90 nitric acid. Thus, to 100g finely divided graphite is added 300g K perchlorate and sufficient amt of 90 nitric acid to yield a liquid mass. The reaction mass is allowed to stand for 4–6 days with nitric acid renewed 4–5 times as it is absorbed. The product, graphigen, is dried at RT and stored in the dried state at RT. Before use in a rocket, the graphigen is soaked in 35% C_6F_6 for several hrs. Et_3Al in a glass-lined jet container under argon pressure sprayed on the graphigen simultaneously with Amm chloride in liquid ammonia is the other component of the hypergolic system

Ref: S.L. Ruskin, USP 2944881(1960) & CA **54**, 25832(1960)

Graphite, Dry (for Use in Ammunition). Its requirements and tests are in US Military Specification MIL-G-155A of 20 Sept 1962 with Amendment 2 of 13 July 1967, superseding JAN-G-155 of 13 Dec 1944

Graphites shall be of the following grades:
Grades I & III – manufactured
Grades II & IV – natural
Grades I & II are intended for use as lubricants in pelleting expls
Grades III & IV – as glazing agents

3. REQUIREMENTS:

See Table 1 on following page

3.1 Appearance (applicable to Grade II and IV only). Graphite of grades II and IV shall appear steel gray or silver gray in color and shall have a metallic luster

3.2 Properties. Graphite shall conform to the limits for the properties specified in Table I, when determined as specified in the applicable sub-paragraphs of 4.3

3.3 Lubricating quality (applicable to Grade I and II only). When specified, Grade I and II graphite shall be tested for use as a pelletizing lubricant as described in paragraph 4.2.2.1

Table I

Properties	Grade I percent	Grade II percent	Grade III percent	Grade IV percent	Paragraph
Moisture, maximum	0.20	0.50	0.50	0.50	4.3.1
Ash, maximum (max)	0.60	6.0	0.60	6.0	4.3.2
Silica, max	–	1.0	–	2.75	4.3.3
Other grit	None	None	None	None	4.3.4
Acidity	None	None	None	None	4.3.5
Free sulfur, max	0.02	–	0.05	0.05	4.3.6
Total sulfur, max	0.20	–	0.50	0.50	4.3.7
Granulation:					
Thru US standard sieve No 100 (149 micron) min	–	65.0	–	–	
Thru US standard sieve No 200 (74 micron) min	96.0	–	96.0	–	
Thru US standard sieve No 325 (44 micron) min	–	–	–	96.0	

3.4 Glazing quality (applicable to Grade III and IV only). When specified Grade III and IV graphite shall withstand the glazing test specified in paragraph 4.2.2.2

4. QUALITY ASSURANCE PROVISIONS

4.1, 4.2, 4.2.1 & 4.2.2 – See p 2 of Spec

4.2.2.1 *Pelletizing Quality (Applicable to Grade I and II).* Mix two parts by weight of the sample and 100 parts by weight of Tetryl, complying with the requirements of Specification JAN-T-339. Make at least 25 pellets of the mixture by subjecting portions of the mixt to pressure in a pelletizing machine *Note:* The graphite shall be considered satisfactory if the machine works smoothly and easily, if the powder does not stick to any parts of the machine, and if the resulting pellets do not fall apart

4.2.2.2 *Glazing quality (applicable to Grade III and IV).* The graphite shall be subjected to an actual factory glazing operation and to such additional tests as shall be considered necessary by the contracting officer

4.2.3 and 4.2.4 – See pp 3.4 of Spec

4.2.5 *Testing.* The lot shall be sampled and specimens selected for testing by the following procedure:

4.2.5.1. *Sampling.* Select 10 percent of the containers in the lot. If there are less than 100 containers in the lot, select 10 containers. If there are less than 10 containers in the lot, all containers shall be selected. Remove a portion of aporox 2 oz of material from each container. Mix thoroughly each of the primary samples so obtd and remove sufficient material to form a composite sample of approx 20 ounces. Mix the composite sample thoroughly, place this in an air-tight container, and label so as to show the name of the material, manufacturer, plant, contract or order number, lot number and number of pounds in the lot. All acceptance tests shall be made on the composite sample representative of the lot. If the composite sample fails to comply with any of the requirements specified, the lot shall be rejected

4.3 *Test Methods and Procedures.* The following test methods and procedures shall be performed

4.3.1 *Determination of Moisture.* Transfer to a tared glass weighing dish a portion of approx 2 grams (g) of the sample and weigh accurately. Dry the dish and contents at 100°C to 105°C for 1 hr, cool in a desiccator and weigh. Calculate the loss in weight and percent of moisture

$$\text{Percent moisture} = \frac{100A}{B}$$

where:

A = loss in wt of dish & specimen

B = wt of specimen

4.3.2 *Determination of Ash.* Transfer (to a tared, porcelain crucible for Grade I & III, and platinum crucible for Grade II & IV) a portion of approx one g of the sample and weigh accurately. Using a gas flame or muffle furnace, ignite the crucible and contents until all combustible matter has been removed, cool

in a desiccator and weigh. Retain the residue for the silica and other grit detns as specified in 4.3.3 and 4.3.4. Calculate the wt of residue as percent ash as follows:

$$\text{Percent ash} = \frac{100\,(A)}{B}$$

where:

A = wt of residue
B = wt of specimen

4.3.3 *Determination of Silica (Applicable to Grade II & IV only).* To the ash in the platinum crucible, add 5 ml of HCl and a few drops of sulfuric acid. Evaporate and ignite. Cool in a desiccator and weigh. Calculate the loss in weight as percent silica as follows, and retain the residue for grit detn:

$$\text{Percent silica} = \frac{100\,(A-C)}{B}$$

where:

A = wt of residue as found in 4.3.2
B = wt of specimen
C = wt of residue after treatment

4.3.4 *Determination of Other Grit*

4.3.4.1 *Applicable to Grade I & III only.* Tranefer at least three portions of approx 0.1g each of the sample to smooth glass slides. Rub the material between glass slides determining the presence of grit by scratching noise and scratches on the glass slide

4.3.4.2 *Applicable to Grade II & IV only.* Transfer the residue from silica detn to a smooth glass slide. Rub the material between glass slides and detn the presence of grit by scratching noise, and scratches on the glass slide

4.3.5 *Determination of Acidity.* Transfer a portion of approx 10g of the sample of a 250-ml beaker, add 100ml to neutral distd water and heat quickly to boiling while stirring. Filter immediately and cool to RT. Test the filtrate by adding two drops of phenophthalein and then two drops of methyl red. If colorless to phenolphthalein and yellow to methyl red, acidity may be reported as none

4.3.6 *Determination of Free Sulfur.* Extract an accurately weighed portion of approx 25g of sample with 50ml of the boiling ethyl ether for approx 15 mins. Filter, wash the residue with ether, and evaporate the combined filtrate and washingd to dryness. To the ether sol residue add 10ml of concd nitric acid, 5ml of concd HCl and again evaporate the mixture to dryness. Moisten the residue with 2–4ml of concd HCl and then add 30–40ml of hot distd water. Filter, wash the residue with distd water, keeping the final volume of filtrate and washings within 100ml. Heat the filtrate to boiling and rapidly add 10ml of 5% barium chloride soln with vigorous agitation, and allow the barium sulfate ppt to settle for at least 24 hours. Filter thru a tared fine porosity crucible, wash the residue with hot distd water and test the last few washings of the ppt with 2 drops of silver nitrate to see that all chloride is removed. Ignite at dull red heat, cool in a desiccator and weigh. Calculate the increase in weight, corrected the barium sulfate obtd in a blank detn on all reagents, to percentage of free sulfur in the specimen as follows:

$$\text{Sulfur percent} = \frac{13.73\,(A-B)}{W}$$

where:

A = wt of ppt in sample
B = wt of ppt in blank
W = wt of specimen

4.3.7 *Determination of Total Sulfur.* Transfer an accurately weighed portion of approx 1g of the sample to a beaker. Add 30ml of concd nitric acid and 0.1g of the sample to a beaker. Add 30ml of concd nitric acid and 0.1 of sulfate-free sodium carbonate. Heat the mixt to boiling and then add 60ml of concd HCl, slowly at first, until violent reaction has subsided. Evaporate the soln to dryness. Moisten the residue with 2 to 4ml of concd HCl, add 30–40ml of hot distd water and digest for a few mins on a steam bath. Filter the mixt and wash the residue with distd water, keeping the final volume of filtrate washing within 100ml. Heat the filtrate to boiling and rapidly add 10ml of 5% barium chloride soln, with vigorous agitation. Allow the ppt of barium sulfate to settle for at least 24 hours. Filter the mixt thru a tared fine porosity crucible, wash the residue with hot distd water and test the last few washings of the ppt with 2 drops of silver nitrate to see that all chloride is removed. Ignite at dull red heat, cool in the desiccator and weigh. Calculate the increase in weight, corrected for the barium sulfate obtd in a blank detn of all reagents, to percentage of total sulfur in the specimen as follows:

$$\text{Sulfur percent} = \frac{13.73\,(A-B)}{W}$$

where:

A = wt of ppt in sample
B = wt of ppt in blank
W = wt of specimen

4.3.8 *Determination of Granulation.* Place an accurately weighed portion of 25g of the sample and two metallic washers on the appropriate US standard sieve, in accordance with Specification RR-S-366, assembled to a bottom pan. Place a cover on the sieve and shake the assembly for 10 mins by hand or 5 mins by means of a mechanical shaker geared to produce 300 + 15 gyrations per min and 150 + 10 taps of the striker per min. When no more material passes thru, weigh the portion retained onthe sieve and calculate the percentage of specimen passing thru the sieve as follows:

$$\text{Percent thru} = W - \frac{(A \pm B)\,100}{W}$$

where:

A = wt retained on designated sieve
B = wt retained on sieves nested above designated sieve
W = wt of specimen

5. PREPARATION AND DELIVERY. See Spec, p 6

6. NOTES. See Spec, pp 6 & 7

Graphite Determination in Propellants. When graphite is present alone, it can be detd by one of the procedures briefly described in Vol 2 of Encycl, p C57-R. It is essentially the same as that of MIL-STD-286A, Method 308.1.2. It is a gravimetric method using concd nitric acid as oxidizer. Parpaillon et al (Refs 1 & 2) prefer using concd hydrogen peroxide in alkaline soln instead of nitric acid. They came to that conclusion after learning that Drs Ernst and Walter Berl decompd nitrocompds by means of 30 hydrogen peroxide in alk soln. Parpaillon & Rigal slightly modified the method of the Berls and applied it to detn of graphite or carbon black in proplnts. This method is described in Vol 2 of Encycl, p C58-R. The Parpaillon method, in the opinion of Mr N. Liszt of Picatinny Arsenal, does not distinguish betw carbon black and graphite and is not as useful. It is also more involved

Mr Liszt sent (Ref 4) the procedure which, in his opinion, is probably the best general method for proplnts and has the advantage of also determining carbon black if it is present. Method 308.1.2 of MIL-STD-286A has serious deficiencies and differences and must be changed in the future editions to conform with the following method 308.1.2 of X-PA-PD-1219, which was developed at PicArsn

Determination of Graphite and Carbon Black by the Method Recommended by Mr N. Liszt of Picatinny Arsenal

1.1 *Carbon Black Absent.* Transfer a weighed portion of approx 10g of the sample to a 400-ml beaker, add 75ml of approx 70% nitric acid, and heat the mixt on a steam bath. When the reaction starts, remove the beaker and allow the reaction to proceed to completion. The rate of decompn can be controlled, if necessary, by adding a little distd water from time to time. After evolution of oxides of nitrogen ceases, heat the material if all the particles have not been completely decomposed. Cool the beaker and contents, and filter the soln thru a previously ignited Gooch crucible prepd with an adequate layer of asbestos which has been washed with 10 to 15ml of approx 70% nitric acid and then with water. Wash the residue with hot water, remove the filtrate which contains nitric acid, (CAUTION: Do not mix acetone washings with nitric acid soln) and wash with acetone until all the organic material is dissolved as shown by the absence of coloration of the washings. Aspirate until the odor of acetone is no longer discernable. Dry the crucible and contents at 120–130°C for 1 hour, cool in a desiccator and weigh. Ignite the crucible and contents in a muffle furnace until all carbonaceous matter has been burned off, cool in a desiccator and weigh. Calculate the loss in weight of the crucible to percentage of graphite in the propellant on a total volatiles and added constituents-free basis

1.2 *Carbon Black Present.* Proceed as directed in paragraph 1.1 to the point where the crucible and contents have been dried at 120–130°C cooled in a desiccator and weighed. The contentents of the crucible represent the weight of the carbon black plus graphite. Place the crucible plus the carbonblack-graphite residue in a flat bottom, straight wall flask. Add 5ml

of cp 70% nitric acid to the crucible and 45ml of the acid to the flask. Connect the flask to a water cooled condenser and heat the contents of the flask to the boiling point by means of a hot-plate. Boil for 3 hours; allow the flask to cool, remove from condenser, remove the crucible, place in a crucible holder and filter the contents of the flask thru the crucible with strong suction. Wash the crucible and contents several times with distd water. Discard the filtrate and washings and continue to wash the residue in the crucible with water and acetone in order to remove all undissolved carbon black as may be determined by the absence of color in the washings and also the appearance of the remaining residue. Give several final washes with acetone; remove the crucible and place in an oven. Dry for 1 hour at a temp of 120–130°C. Remove, cool for 30 mins in a desiccator and weigh. Calculate the loss in weight from the previous weighing of the crucible plus residue to percentage of carbon black as follows:

$$\text{Percentage of carbon black} = \frac{100A}{W}$$

where:

A = wt of crucible + carbon black residue-loss of wt of crucible after 70% nitric acid boil

B = wt of sample

Place the crucible in a muffle furnace at a temp of 750–800°C and permit to remain until all carbonaceous matter has been ignited, Remove from furnace; cool for several mins in the atmosphere; place in a desiccator; cool further for 30 mins and weigh. Calculate the loss in weight to percentage of graphite as follows:

$$\text{Percentage of graphite} = \frac{100A}{W}$$

where:

A = loss of wt of the crucible after ignition of 1g

W = original wt of the sample

A similar method for determination of graphite and carbon black is described in Std Methods of Chem Analysis (Ref 3) under the title "Carbon Black and Graphite, Gravimetric". It is not included here because it is assumed that every chem lab has a copy of Std Methods

Refs: 1) M. Parpaillon, MP **34**, 419–20 (1952) 2) M. Parpaillon & S. Rigal, MP **37**, 305–17 (1955) & CA **50**, 17451 (1956) 3) StdMethods-ChemAnalysis, Vol **2**, Part B (1963), p 1378 4) Nathan Liszt of Picatinny Arsenal; private communication (1974)

Graphite, Pyrolytic or Pyrographite. A dense graphite, stronger and more resistant to heat than ordinary graphite, and is used in rocket nozzles, missiles in general, and nuclear reactors. It can be prepd by a recrystn process from ordinary graphite. For this, forms of graphite are heated in a stream of a hydrocarbon gas. The C in the gas is deposited on the original form with the C crysts in alignment along the flat planes of the form. The pyrographite has a high tensile strength even at 5000°F. Sheets only one mil thick are impervious to liquids and gases. Its destruction temp is ca 6600°F

Refs: 1) CondChemDict (1961), 550-R & 551-L and (1971), 426-R 2) Kirk & Othmer, 2nd edit **4** (1964), 158ff (Carbon, Graphitized)

Graphitic Oxide (Graphitic Acid), $C_7O_2(OH)_2$; mw 150.09; a solid substance of variable color ranging from green to brown, prepd by treating graphite with strong oxidizing agents, such as nitric acid with K chlorate or mixed acid (nitric + sulfuric acid) with K nitrate (Ref 2, p 828 & Ref 4, p 437). When oxidation with nitric acid is conducted in presence of a catalyst, such as vanadic acid or silver nitrate, the resulting product is mellitic acid (Ref 8)

Brodie (quoted in Refs 2 & 7) assigned the formula $C_{11}H_4O_5$ to the first compd, but this has now been disproved. The compd was originally called "acid" because of the ease in which it dissolved in alkalies

More recently the product was investigated by K.A. Hofmann, U. Hofmann and others (Refs 3, 4, 5 & 6) and was assigned a very complicated structural formula (Ref 6, p 23 & Ref 7, p 459)

The product is now called "graphitic oxide" and has a varied compn with a ratio of carbon to oxygen of 2.9-3.5:1, and which can be approx distinguished by the color of the substance

Karrer (Ref 8, p 520) considers graphitic

acid as an intermediate product obtd in oxidation of graphite to mellitic acid (Ref 8)

When heated, graphitic oxide decomp almost explosively (Ref 7)

Refs: 1) V. Kohlschutter, ZAnorgChem **105**, 121(1918) 2) Mellor **5**(1924), 828 3) K.A. Hofmann, Ber **59**, 2433(1926) 4) U. Hofmann, Ber **61**, 435(1928) 5) U. Hofmann & A. Frenzel, Ber **63**, 1248(1930) 6) U. Hofmann, A. Frenzel & E. Csalán, Ann **510**, 1–41(1934) 7) H.J. Emeléus & J.S. Anderson, "Modern Aspects of Inorganic Chemistry", VanNostrand Co, NY (1942), pp 457–59 8) Karrer (1947), 520 9) Kirk & Othmer, not found 10) CondChemDict (1971), 426-R

Graphitic Oxide By Explosion. Carbon prepd by the expln of graphitic oxide has a different structure than any other form of C. Studies with the electron microscope and with X-rays and electron absorption indicate that it consists of very thin graphite layers which show the property of increasing specific resistance with increasing pressure. From detns of the methylene blue adsorption and the catalytic activity in synthesizing HBr, it is concluded that the crystal faces are active rather than only the unsatd atoms in the edges of the faces

Ref: G.L. Ruess & F. Vogt, Monatsh **78**, 222–42(1948) & CA **42**, 7126h(1948)

Graphitic Oxide Explosives. Mixtures of "graphitic oxide" with MF, TNT, PA were patented for use in priming compns

Ref: A.W. Schorger, USP 1681259(1928) & CA **22**, 3780(1928)

Graphiting of Smokeless Propellants, also known as **Glazing** (qv). French method, called *plombinage* or *lissage* is described by Pepin Lehalleur (1935), p 306. Amer method is described in Davis (1943), p 306. The "sweetie barrel" used for tumbling proplnt grains with graphite is illustrated on p 291, Fig 71. This apparatus is also known as "tumbling barrel", "double-cone mixer" and "mushroom mixer" and is shown as Fig 3 on p D1558 of Vol **5** of Encycl

Gravimetric Analysis, Inorganic. That branch of quantitative chemical analysis in which a desired constituent is converted (usually by precipitation) to a pure compd or element of definite, known compn, and is weighed. In a few cases, a compd or element is formed which does not contain the constituent but bears a definite mathematical relationship to it. In either case, the amount of the desired constituent can be detd from the weight and compn of the precipitate. Methods exist for the detn of all the elements by gravimetric analysis

Gravimetric analyses of inorg expls and components of expl compns are described individually

Refs: 1) F.E. Beamish, AnalChem **21**, 144–60(1949) (Review of inorg gravimetric analysis with 362 refs) 2) L. Erdey, "Gravimetric Analysis", English translation from Hungarian by G. Svehla, Macmillan Co, NY, Vol **7**, Part II (1965)

Gravimetric Analysis, Organic. It concerns itself with carbon and hydrogen and various compds of these. Hydrocarbons, aldehydes and ketones, acids and derivatives, amines and related compds, sulfur-contg compds, phenols and derivs, and natural products such cholesterol are analyzed using these methods. Many of the org compds contg nitrogen, such as azides, diazo, nitrites, nitrates and nitrocompds, aliphatic and aromatic are expl and their analytical procedures are usually described individually. Many org compds contg peroxide group also are expl and their analyses are described individually

Refs: 1) J.F. Flagg, AnalChem **21**, 160–63 (1949) (Review on org gravimetric analysis; included 51 refs) 2) L. Erdey, "Gravimetric Analysis", English translation from Hungarian by G. Svrhla, Macmillan Co, NY, Vol **7**, Part I (1963)

Gravimetric Density or **Apparent Specific Gravity.** See Vol **3** of Encycl, p D66, under DENSITY

Gravimetric Density of a Propellant. Accdg to US OrdnProof Manual **7-26**(1945), pp 31–2,

gravimetric density (gr d) represents a composite measurement of its relative bulk and flowing qualities. For its detn, the grains of proplnt are allowed to flow thru a restricted orifice of constant diam (½ inch), under the pressure of a given head (or quantity of proplnt) and to drop a distance of 7 inches into a measuring cup, which can hold 324.9±0.1ml of water at 20° (which is equivalent to 5000 grains). On dividing the wt of proplnt in the cup by the wt of w, the value of gr d is obtd. Because of the method of measurement used, the gr d is not related directly to sp gr. Thus, two proplnts of the same sp gr but of different granulation, or having dissimilar surface conditions, would not flow nor pack the same, and as a result, would not have the same gr d

In general, the gr d indicates the relative quantity of proplnt that can be loaded into a cartridge case. Other things being considered equal, the higher the gr d, the greater the wt of proplnt that can be charged into any given volume and, conversely, the lower the gr d, the lighter the wt of the charge

Grave, I.P. A Russian ballistician, born in the 1870's, who started to teach in the 1900's at the Imperial Artillery Academy at St Petersbourg. He published in 1932–38 books on "Interior Ballistics" in five volumes, which may be considered as an Encyclopedia of Ballistics. He was still active in 1940 as professor in Artillery Academy. He developed in 1916 a type of smokeless proplnt suitable for rockets, but its application was postponed till 1928
Ref: M.E. Serebriakov, "Vnutrenniayia Ballistika Stvol'nykh Sistém i Porokhovykh Raket" (Interior Ballistic of Gun Barrel Systems and Solid Propellant Rockets", Oborongiz, Moskva (1962), pp 15, 19 & 23–5. Abbreviated Engl translation by Dr V.A. Nekrassoff, formerly of Aberdeen Proving Ground, Maryland

Graydon Explosive. Mining expl prepd by impregnating sheets of cotton or wool textile fabrics with NG and then placing them betwn two sheets of paraffined paper. The combined sheets were rolled into a solid cylinder with diam to fit the bore-hole, and then cut to the proper length

Refs: 1) Daniel (1902), 35 2) Fedoroff & Clift, Vol 4 (1946), p 41

Greaves and Hahn Explosives. BkPdr, Cordite or NG Expls to which oxalates (such as of ammonia) were added. They were introduced in 1895 in England, but it seems that Schöneweg had already proposed similar expls in 1886 in Germany
Refs: 1) Daniel (1902), 357 2) Fedoroff & Clift 4 (1946, 41

GrBlP (Ger abbr for Grobes Blättchenpulver). A large-flake, single-base, smokeless proplnt used during WWI in Ger 150 and 210mm cannons
Ref: Marshall 1 (1917), 303

Greek Fire. See under Black Powder in Vol 2, p B166-L. See also Fire, Greek in this Vol, p F31-L
Ref: J.R. Partington, "A History of Greek Fire and Gunpowder", W. Heffer, Cambridge, Engl (1960)

Green Cross (Grün Kreuz). A German term for lung-injurious agents used as offensive gases
Ref: Wachtell (1944), 99

Greener (Poudre). Smokeless propknt based on NC, to which could be added NBz, graphite or carbon black. It was imported to England
Ref: Daniel (1902), 357

Greener, William (1806–1869). British expert on guns who published in 1835 the book entitled "The Gun" and in 1841, "The Science of Gunnery", which, besides embodying the gun, dealt with cannon. He manufd guns beginning in 1829 at Newcastle and then in 1844 transferred his business to Birmingham. He introduced many improvements in gunnery. Before his death in 1869 his business went to his son, known as **W.W. Greener** (1834–1921) who also became an expert in gunnery. He became known for improving and introducing the so-called "choke-bore", invented in 1874 in the

USA. He published in 1881 the book: "The Gun and Its Development". The 9th enlarged edition (304pp), published in 1910, is in possession of authors of this Encycl. He has written since 1871 a total of seven books, some of which have reached nine editions. His books were translated into many foreign languages
Refs: 1) Introduction to the 9th Edition (1910) of the book of W.W. Greener, "The Gun and Its Development", Bonanza Books, New York 2) Encyclopedia Britannica **10** (1973), 894

Greenhill, Sir George (1847–1927). A British ballistician who was the Director of the Ballistic Research Dept, Woolwich
Ref: A.D. Crow, MAF **7**, 429–34 (1928)

Green Powder (French Anarchist's Explosive). During the assaults conducted by these revolutionists, the following expl of the Cheddite type was used in some bombs: $KClO_3$ 49, $K_4Fe(CN)_6$ 28 & Sugar 23. Due to its extreme sensitivity, the expl was dangerous to handle
Refs: 1) Pérez Ara (1945), 222 2) Giua, Trattato **6 (1) (1959), 396**

Gregorini's Explosive. AN 80, K nitrate 5 & Al 15 to which was added 10% of a mixture of MNT 75, glycerol 5, Collodin 15 & $K_2Mn_2O_8$ 5%
Ref: G.A. Gregorini, BritP 1237 (1911) & CA **6**, 1991 (1912)

Gremuchaya Rtut' ili Ful'minat Rtuti. Russ for Mercuric Fulminate, which is still used in Russia for loading some blasting caps (See also under FULMINATES in Vol **6**, pp F216ff
Refs: PATR **2145** (1955), p Rus 8 2) Gorst (1957), 108 and (1972), 99

Gremuchii Studen'. Russ for Blasting Gelatin, contg NG 92 & NC 8%
Ref: Yaremenko & Svetlov (1957), 183

Grenade. Fr for Grenade, such as Grenade à fusil: Rifle Grenade; – à main: Hand Grenade; – fumigène: Smoke Grenade; – fusante: Time Grenade; – incendiaire: Incendiary Grenade; – à manche: Stick Grenade; and – suffocante: Asphyxiating Grenade
Ref: Patterson's French-English Dictionary (1921), p 173

GRENADES. A grenade is a small explosive or chemical missile, originally designed to be thrown by hand, but now also designed to be projected from special grenade launchers usually fitted either to rifles (or carbines) or guns ("Gun Grenades", Stielgranat in German).
History. While "rifle grenades" were not introduced until **WWI**, "hand grenades" existed as early as the 15th century. The first grenades were in the shape of a pomegranate and from this resemblance the name originated. By the middle of the 17th century, special military units were organized consisting of tall, strong soldiers called "Grenadiers", who could throw the grenades quite a distance. The grenades used at that time consisted of hollow cast iron balls (spheres) about 2½ inches in diam, contg a charge of BkPdr and provided with a fuse or a slow match. Before throwing the grenade, the fuse was ignited and it took several seconds before the flame reached the charge of the grenade and exploded it

Beginning with the middle of the 18th century, the grenade practically disappeared as a weapon, due to the great progress made at that time in small arms and cannons

The special conditions prevailing at Port Arthur and at the battle of Mukden in the Russo-Japanese war (1904–1905) led to the revival of the hand grenade. A very successful grenade was invented by Capt Lischin. It was a cylindrical grenade charged with dry Guncotton and had a wooden handle attached for convenience in throwing. In Fig copied from Marshall **2** (1917), 566: A is an iron case, C–Guncotton with detonator, and D–a cup which could be removed permit the insertion of detonator and could be turned so as to make the grenade either live or safe. As a further precaution the cap K was kept on until the grenade was used. In more recent Lischin's bombs, the cap K was re-

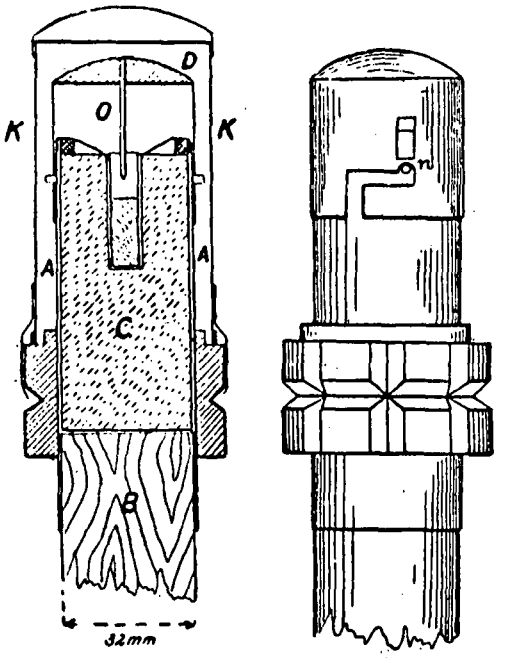


FIG. Lischin's Hand-Grenade Truncheon Type

placed by safety pin and TNT was used instead of Guncotton with an "exploder" betw detonator and the chge. On impact, the pin O was driven into the detonator and fired it. The wooden handle B was ca 18 inches long and had attached to it a tail of dome fabric to cause the grenade to travel nose first

A simpler hand grenade was used by the Russians at the siege of Port Arthur. This *improvised* grenade was prepd by filling discarded cartridges with Ruck-a-rock [See PATR **2145**(1955), p R9-L]

Accdg to Marshall **3**(1917), p 565, the *Hale Handgrenade*, also known as "policeman's truncheon" was similar to Lischin's. In its later model the container was of strong cast steel. Its HE chge was not specified

During WWI the stabilization of the western front and the resort to trench warfare demonstrated the value of the grenade for close fighting, when used against personnel, obstructions, machine gun nests etc. The Germans developed three kinds of grenades: Stielhandgranate, Eierhandgranate and Diskushandgranate. The British developed a very successful *Mills* type grenade (See Fig from Marshall **2**(1917), 566) which was egg-shaped. The French used a cylindrical grenade with a handle. The Americans used both the British and French grenades

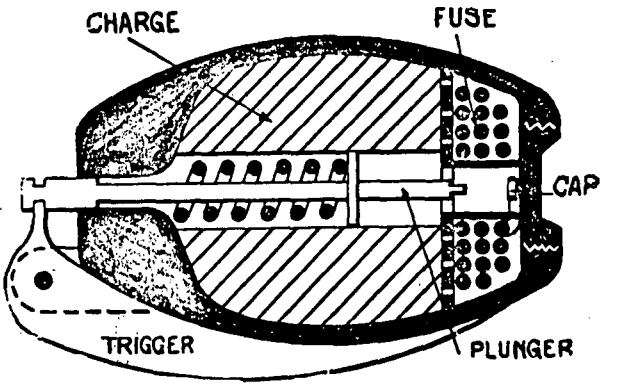


FIG. Mills' Hand-Grenade. Egg Type

and did not bother to develop one of their own

For filling grenades, any kind of HE can be used, provided it is fairly insensitive to shock. Dry NC was one of the first expls used. Other expls used were Ammonals, PA, Cheddites etc . The USA also used a *Grenite* (qv) and another Nitrostarch expl contg NS 25, AN 34, binder 1 & Na nitrate 40%. These were later replaced by EC Blank Powder and finally by TNT

As the range for grenades thrown by hand is rather low (maximum about 35 yards), attempts were made to increase it by using a sling or catapult and, of course, a greater range was attained. With the development of the rifle grenade, ranges up to several hundred yards have been reached. One of the original types of such grenades was *Hale's Rifle Grenade*. This and other grenades intended to be fired from rifles have a rod which goes down the rifle barrel and is fired by a special blank cartridge. A safety pin is removed before firing, but the grenade does not become "live" (armed) until it has travelled some distance, when the striker, which hits the detonator on impact, is released by the rotating motion imparted to the grenade by the vanes attached to it

Since WWI there has been considerable development and standardization of grenades. They may be divided into *fragmentation, demolition or offensive, antitank, smoke, illuminating, chemical* and *practice* grenades

In modern warfare, such as of WWII, the grenade, especially the rifle grenade, has become a diversified weapon. It started to be used not only as an antipersonnel weapon and against machine gun nests, but also against

tanks and armored vehicles, booby traps etc. In addition, it has been used for demolition, for incendiary effect, as smoke producers and for signalling. Progress in grenade design has included greater penetration and larger number of fragments. As fillers, TNT, Pentolite and RDX expls, such as Cyclotols, have been used

Descriptions of grenades used by the US Armed Forces during WWII are given in the book of Maj Ohart (1946), p 354–62). Many of these types of grenades are still in use at the present time (See further)

Grenades used during WWII by the British are listed in **TM 9-1985-1** (1953), entitled "British Explosive Ordnance"; those by the French and Italians in **TM 9-1985-6** (1953); by the Germans in **TM 9-1985-2** (1953) & in PATR **2510**(1958); by the Japanese in **TM 9-1985-5** (1953); and by the Russians in PATR **2145**(1955)

As previously mentioned, there are two types of grenades – hand and rifle. They may be described as follows:

Hand Grenade, as the name implies, is intended primarily to be thrown by the individual soldier. All standard US Army hand grenades have a delay-type fuze except for Grenade M26A2 with fuze M217 which has an impact-delay-type fuze (See Fig 1). Classification accdg to service uses, tactical uses, particular filler and to the time of functioning is given below. Besides their use against the personnel, vehicles (like tanks), and demolition, they can be used for clearing passages thru mine fields

Hand Grenades are kept safe in handling by the safety pin that holds the lever in place, thus restraining a striker under the lever. The lever is grasped by the hand used for throwing, and the safety pin is removed. When the grenade is thrown, the lever flies off, allowing the striker to rotate by spring action and strike the primer. This ignites the delay element of 4–5 secs, after which the grenade functions

Rifle Grenades are designed for projection from a grenade launcher attached to the muzzle of a rifle (See Fig 2). They are used against

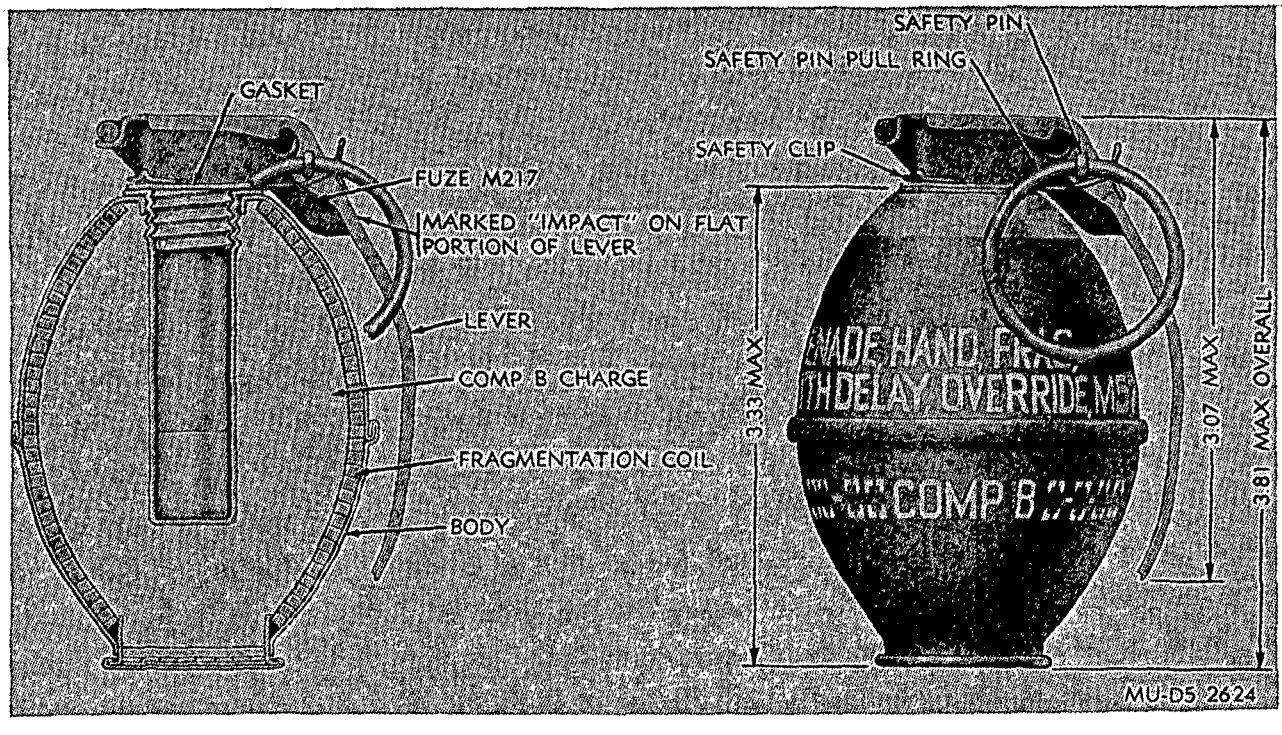


FIG 1 Fragmentation Hand Grenade M57

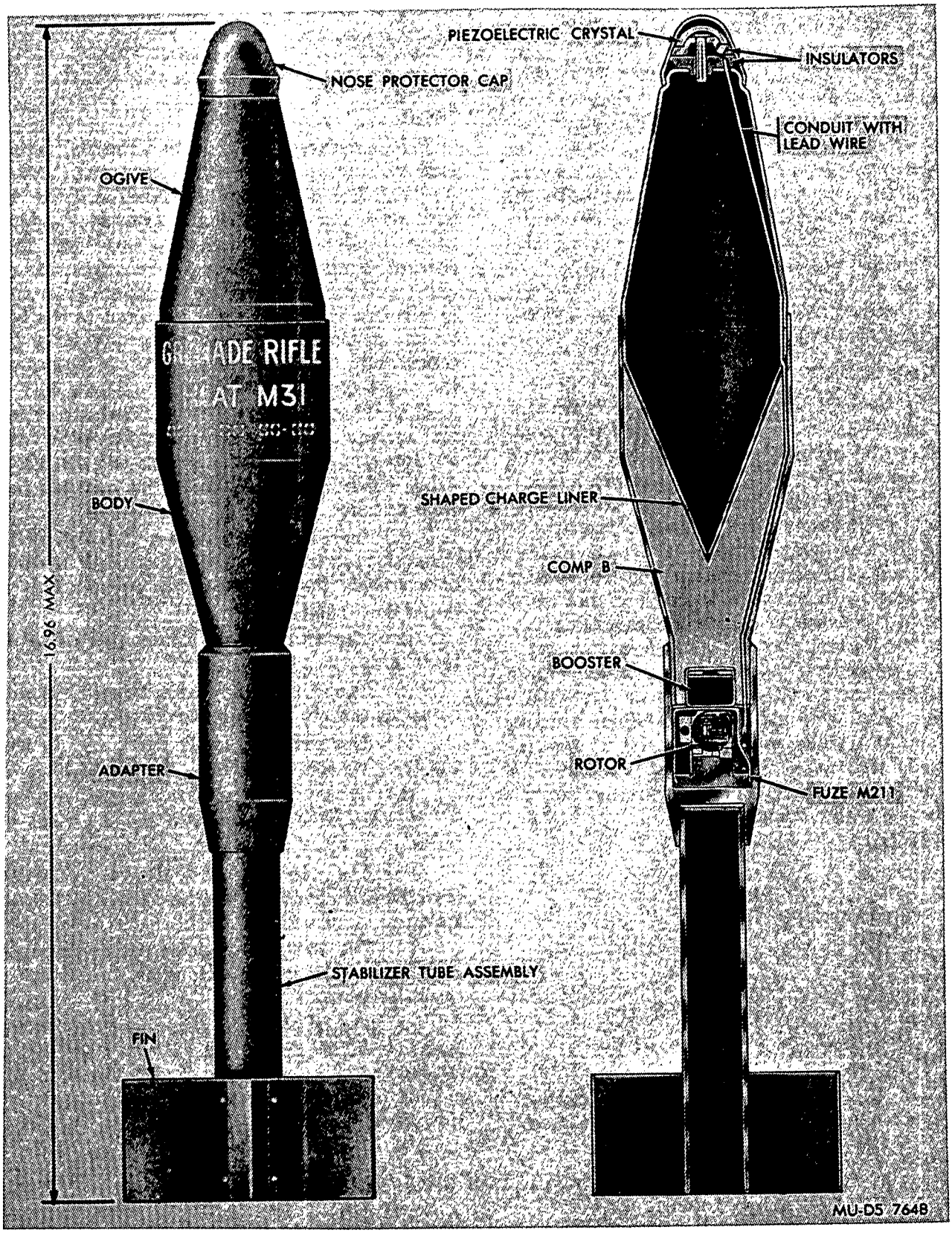


FIG 2 HEAT Rifle Grenade M31

armored targets, against personnel, for screening, signalling, ir for incendiary effect. Like hand grenades, they are kept safe in handling by a safety pin which is removed after the grenade is placed on the launcher and before firing the rifle

Present US hand and rifle grenades can be classified accdg to their **service use** as follows:
Fragmentation Grenades, which contain a HE chge in a metallic body, the fragments of which do most of the damage. The thrower must take cover and cannot continue advancing until a safe time after the detonation. Their effective fragment range is greater than the distance they can be thrown
Offensive Grenades contain a HE chge in a paper body or other thin container and do most of their damage by blast effect, resulting in demolition and lethal shock, rather than by fragmentation. The thrower can, therefore, continue to advance after throwing the grenade which accounts for the name. Their effective fragmentation range is safely less than the distance they can be thrown
Practice Grenades contain a small BkPdr spotting chge to give an indicating puff in practicing with grenades
Training or Dummy Grenades are totally inert and are intended for practice throwing
Note: Accdg to the late A.B. Schilling of PicArsn, Offensive Grenades are designed for use in attack and are so designed as to not impede the advance of the attacker, eg, Base-ball Grenade

Defensive Grenades (usually of the fragmentation type) are used where cover is provided or must be taken by the thrower; eg, Pineapple Grenade

Offensive-Defensive Grenades are so designed that, by the addn of a sleeve or similar device, the offensive design may be used as a defensive type by a fragmentation producing effect

Grenades may also be classified accdg to **tactical use** or accdg to the **particular filler,** as follows:
High-Explosive Grenades contain HE for fragmentation or blast effect, the expl being TNT, Tetrytol, Pentolite, Cyclonite etc
Chemical Grenades contain a special mixture for Chemical Warfare (CW). The special type of filling in common use is HC smoke mixt, and such grenades are known as *smoke grenades*. The smoke mixt may be colored for signalling purposes
Illuminating Grenades contain mixts producing strong light for illuminating terrain (Comp with Flares)
Incendiary Grenades contain an incendiary mixt for starting fires

Grenades may also be classified accdg to the **time of functioning,** as follows:
Automatic Time Grenades contg a fixed delay that is set off automatically when the grenade is thrown or fired from a rifle launcher
Contact Grenades, contg no delay but which function when the grenade hits a target at any distance within range of throwing or launching

Present **US Grenades** are described in **TM 9-1330-200** (1971) entitled "Grenades, Hand and Rifle", Departments of the Army and the Navy, Washington, DC

Hand grenades are of various types which include fragmentation, illumination, chemical & practice. They are of such a size & shape as to be convenient for throwing by hand. Hand grenades are used to supplement small arms against an enemy in close combat, against a variety of combat targets, for riot control, incendiary purposes & training. The five basic types of hand grenades are as follows:
Fragmentation. Used to produce casualties by high velocity projection of fragments
Illuminating. Used to provide illumination of terrain & targets
Chemical. Used for riot control, incendiary, smoke screening, smoke signalling & incapacitating purposes
Offensive. Used for their blast effect
Practice & Training. Used for training personnel in use, care & handling of service grenades

The expls used in fragmentation grenades may be either Comp B or TNT (flaked or granular); illuminating grenades use a pyrotechnic compn; chemical agents CN & CS (See Vol 2, p C167-L) are used in riot grenades; smoke grenades use HC filler (See Vol 2, p C168-L) or SP filler (See Vol 2, p C169-L) or red, green, yellow or violet smoke compn s; incendiary grenades use Thermite & an igniter mixt; TNT (flaked) is used in offensive grenades; and no expl filler is used in practice grenades

Rifle grenades, as the name implies, are fired from a rifle. They may be fired at low angles or high angles, depending on the type of grenade & effect desired. All grenades are stabilized by the use of fins at the rear section. Rifle grenades are used against armored targets, against personnel, for screening or signalling, or as incendiaries against flammable targets. There are three general types of rifle grenades:

Antitank. Used against armored targets or fortification. It contains a Comp B shaped chge capable of penetrating 10 inches of armor plate or 20 inches of reinforced concrete at an effective range of 115 meters

Chemical. There are four basic kinds: WP smoke, HC & colored smoke, incendiary, and smoke streamer. All are used primarily to produce smoke either for signalling or screening effects. The WP smoke also has incendiary capabilities. The smoke grenade & smoke streamer use a chge of baking soda, K chlorate, sugar and a dye to produce green, red, violet or yellow colored smoke

Practice. These grenades use no HE chge. They are used for training personnel in the care, handling & use of rifle grenades prior to training with service grenades. One type is issued ready for use; other types are assembled from practice & training hand grenades and grenade projection adapters

With a grenade projection adapter, a hand grenade can be converted into a rifle grenade. The adapter consists of a stabilizer tube with a fin assembly on one end and gripping claws on the other end

Addnl Refs for Grenades:

A) ArmyOrdnHdb **154**, "History of Trench Warfare Material", USGovtPtgOfc, Washington, Dc (1920), pp 182–214

B) US Army Chief of Ordnance, "Safety and Storage Manual for Explosives and Ammunition", OO Form **5994**, Washington, DC (1928), Sect XXI, pp 1–6

C) A.M. Prentiss, "Chemicals in War", McGraw-Hill, NY (1937), 295–304

D) "Ammunition Inspection Guide", WarDept-TechManual **TM9–1904**, Washington, Dc (1944), pp 752–71 & 776

D_1) M. Knayer, SS **39**, 98–101 (1944) (Ueber die Entwicklung der Granatwerferwaffe)

E) "Small Arms Materiel and Associated Equipment", **TM9–2200** (1949)

F) J.H. Frazer et al, USP 2504119 (1950) & CA **44**, 6130 (1950) (Use of hand grenade to clear land mines after spraying field with a mixt of perchloric acid & ethylene glycol monoethyl ether)

G) J.F.C. Fuller, "A Military History of the Western World", Funk & Wagnalls Co, NY (1955), 49

H) R.C. Schofield, PA Memo Rept **153** (1958) (Examination and Evaluation of Grenade, Hand, Fragmentation, Mod(?) and Fuze. Experimental

I) A. Langhans, Soldat und Technik **1961**, 192; Explosivst **10**, 227 (1962) (Ein neues Granatengewehr)

J) A.B. Schilling, PATM **1129** (1963) (Examination and Evaluation of Grenade, Hand, Defensive, Model PL-61. Austrian)

K) H. Schmolmann, Auslegeschrift 1155694 (1963); Explosivst **12**, 73 (1964) (Handgranate, die als Gewehrgranate verwendbar ist)

L) W.T. McInnis, "Launcher Grenade XM129. Analysis of Stresses Due to Dynamic Loads", Springfield Armory **TR–2741** (Aug 1965)

M) Col J. Crossman, "Grenades, Now and Then", Ordn **51**, 62–66 (1966)

N) Anon, "Grenades, Hand and Rifle, US Army **TM9–1330–200** (1966)

O) R. Bigelow, Newark News Magazine (8 Oct 1967)

P) Anon, "Grenades", US Army Materiel Command Pamphlet **AMCP 706–240** (1967)

Q) E. Luttwack, "A Dictionary of Modern War", Harper & Row, NY (1971), p 102

R) US Military Specifications:

Grenade Launcher, M7A3, MIL-L-1225B (Nov 1957)

Grenade Launcher, M76, MIL-L-45528A (March 1965)

Grenade Launcher, 40mm, M79, MIL-L-45589 (Feb 1970)

Grenade Launcher, 40mm, XM129, MIL-L-45922 (March 1971)

Grenade Launcher, 40mm, Detachable, M203, MIL-L-45935 (Sept 1971)

Grenade Launcher. A device which functions as an extension of the barrel of a rifle which permits firing of rifle grenades. The launcher is secured to the muzzle of a rifle by means of a clip latch which fits over the bayonet stud.

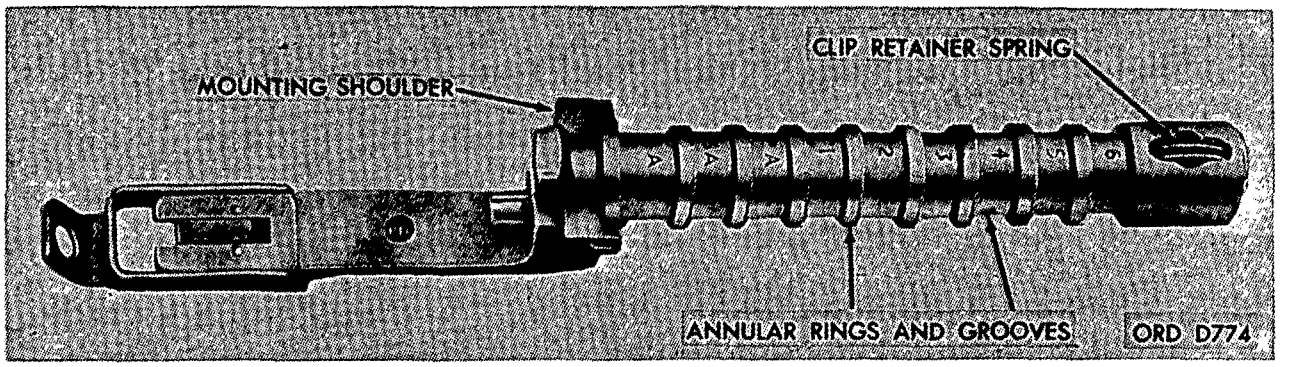


FIG 3 Grenade Launcher , M7A3

Fig 3 is an illustration of the Grenade Launcher M7A3. The M79 Grenade Launcher is a 40mm tube with firing mechanism which can send a grenade as far as 400 meters. Besides HE grenades, it can launch illuminating, smoke and signalling missiles. Developed at Pic-Arsn and used successfully in Vietnam War (Addnl Ref Q)

Cartridges for propelling rifle grenades are briefly described in Vol 2 of Encycl, p C74-R, item c) and illustrated on p C75. More detailed description is given in "Small Arms Ammunition", **TM 9-1305-200**(1961), pp 50-1

Grenades, Special Topics and Testing.

Lots. Grenades are divided into lots of various sizes

Packing. Grenades should be packed separately from bouchons and firing mechanism, each in a separate wooden box or in a separate compartment of the same box. The exception is in the case of grenades loaded with EC Blank Fire Powder which can be packed with firing mechanism assembled

Maintenance and Surveillance. Similar to the regulations outlined under "Trench Mortar Ammunition"

Renovation and Salvage. Similar to regulations outlined under "Trench Mortar Ammunition", with the exception that bouchons and firing mechanisms are not renovated or salvaged unless directed by the proper authority

Destruction. For offensive hand grenades and and rifle grenades, not more than 40 grenades shall be packed in close contact with each other in a box and the ensemble placed in a pit, about 4 ft deep, free from stones or other debris that may form missiles. On top of the pile and in intimate contact are placed several ½ lb TNT blocks, using 1 block for each 6 grenades. One of the blocks in the pile is fitted with a No 8 electric blasting cap, to which is attached several feet of BkPdr safety fuse. A lid should be placed over the box (taking care that the safety fuse is not pinched) and the box should be covered with 3-4 ft of earth or other suitable material

If only a limited quantity of grenades have to be destroyed, it may be done by burning. For this, a pit similar to that used for above-mentioned grenades, is provided with a heavy metal cover to stop flying fragments and unexploded grenades. A fairly thick layer of flammable material, such as wood or coke, is placed under the pile of grenades and, as soon as the fire is started, the cover is placed over the pit and all persons retire behind a barricade

For destruction of "fragmentation" grenades, the following method may be used:

After unscrewing the fuze from the body of the grenade, the EC Blank Fire Powder is emptied into a barrel of water and the fuze is destroyed by the "pit and chute" method, as described under "Metal Components, Destruction". The EC Blank Powder may later be spread out on the ground, allowed to dry and burned, as described under Black Powder

If fuzes cannot be disassembled easily, the grenades should be destroyed with demolition blockd in the same manner as offensive and rifle grenades

Safety Precautions. Grenades and bouchons should never be dropped, thrown or subjected to rough handling. No tampering should be allowed, as many accidents have been due to this cause

Testing of Grenades. For *hand grenades* the test consists in projecting them from a pneumatic "grenade thrower" (which removes the safety pin from the fuze simultaneously with the opening of the air valve of the grenade propelling apparatus) and making the following observations:

a) The time from the instant of propelling until detonation

b) The order of functioning

c) The promptness of the release of the handle

For *rifle grenades* the test is conducted essentially in the same manner, with the exception that the grenade is fired from a rifle instead of being propelled from a pneumatic grenade thrower. The "antitank grenades" are usually fired against an armor plate

All personnel must be under cover during testing of hand & rifle grenades

Refs: 1) Anon, "Ordnance Proof Manual" **OPM 12-10**(1943) 2) Ditto, **OPM 10-90** (1957) 3) US Army Materiel Command, "Safety Manual", **AMCR 385-100** (April 1970), p 28-15

Grenadine (or Granatina). Mining explosive patented in 1882 by Sala and which contained K nitrate, sulfur, benzene, glycerin, ashes and sand

Refs: 1) Daniel (1902), 361 2) Fedoroff & Clift **4**(1964), 4

Grenadine. Mining explosive patented in 1883 by Gacon: K nitrate 69, sulfur 19, ashes and tannin mixed with water 12

Ref: Daniel (1902), 361

Grenage. Fr for Graining

Grenée Poudre. French smokeless proplnt manufd at Moulin Plank: NC 60, Ba nitrate 30, K nitrate 6, Gelose (agar-agar) 3, paraffin 1%

Refs: 1) Daniel (1902), 381 2) A. Pérez Ara (1945, 440 (Pólvora Grenée)

Grenite (Amer). A NS expl of varied compn: NS 95.50–98.25, petroleum oil 0.75–2.00, gum arabic 0.75–2.00, moisture 0 to 1.00%. Was used during WWI in hand grenades. Its explosive properties have been experimentally determined. See also Vol **5**, under DYNAMITE, class X, item (d), p D1611-L

Refs: 1) Davis (1943), 275–76 2) A. LeRoux, MP **33**, 211–21 (1951) & CA **47**, 10229 (1953)

Gribeauval, Jean-Baptiste de, (1715–1789), French General who completely revolutionized artillery. This quarter century later contributed so much to Napoleonic victories

Refs: 1) "Petit Larousse Illustré", Paris VI (1953), 1415-L 2) R.E. Dupuy & T.N. Dupuy, "The Encyclopedia of Military History", Harper & Row, NY (1970), 665. Numerous illustrations on pp 738–39

Gricellite-couche or **Lignamite-couche** (Fr). Permissible expl contg NG 12.0, CC 0.5, AN 86.0 & cellulose 1.5%

Ref: Vennin, Burlot & Lécorché (1932), 554

Griess, Peter (1829–1888). German chemist, discoverer of the diazotization reactions which are of very great importance in the dye industry. These reactions serve also in the prepn of some expls, as for example Diazodinitrophenol (DADNPh), described in Vol **2** of Encycl, p B59-L as Dinitrobenzenediazoöxide. Its tests are given in Vol **5**, pp D1160-R to D1163-L

Refs: 1) A.W. Hoffman, E.Fischer & M. Caro, Ber **24**, 1008–76, I–XXXVIII (1891) 2) Hackh's Dict (1944), 389-L 3) Sister U. Heines, JChemEd **35**, 187–91 (1958) (Peter Griess-Discoverer of Diazo Compounds)

Griffing's Explosives. 1) Granulated mixt of sugar 25, K chlorate 30, powdered charcoal 15, powdered MnO_2 15, milk sugar 5 & Zn flue dust 10% (Ref 1) 2)$KClO_3$ 50, sugar 49, $KMnO_4$ 0.34, S 0.33 & talcum powder 0.33% claimed to be suitable as Gunpowder (Ref 2)

Refs: 1) C.E. Griffing, USP 991057 (1911) & CA **5**, 2332 (1911) 2) C.E. Griffing, USP 1282413 (1918) & CA **13**, 187 (1919)

Grigia (Ital). Older blasting expls: NG 20–25 & BkPdr 80–75%
Ref: Giua, Trattato VI(1)(1959), 340

Grignard, Victor (1871–1935). French chemist, receipient (with Paul Sabatier) of Nobel Prize in chemistry (1912). Discovered organo-magnesium compds used for organic synthesis (See Grignard's Reaction and Reagent)

Author of monumental work: "Traité de chimie organique", 15 vols (1935)
Refs: 1) Hackh's Dict (1944), 389-L 2) A.G. Debus, Edit, "World Who's Who in Science", Marquis Who's Who, Inc, Chicago, Ill (1968), 705 3) Encyclopedia Britannica 10(1973), 926

Grignard's Reaction and Reagent. *Grignard's reaction*, also known as magnesium alkyl condensation, serves for introducing by means of G's reagent of a carbon atom into the hydrocarbon radical of a compd. It offers a ready means of passing from a lower member of a homologous series to a higher member. *Grignard's reagent* is any of a group of organic magnesium compds of the general type RMgX, where R is an org radical and X is a halogen (usually iodine)
Refs: 1) Hackh's Dict (1944), 389-L & R 2) Encyclopedia Britannica 10(1973), 926–29

Grignon. Spanish for the residium left after pressing the oil from olives. When distilled it yields ca 4% Ca acetate and 1.2% of crude wood alcohol
Note: If the distn does not go to carbonization the residue is a cellulose and there is no reason why it cannot be nitrated to produce NC. If the residue is carbonized, the resulting product should be suitable as an ingredient of expls in lieu of charcoal. This, of course, concerns only the countries with large production and consumption of olives and short supply of cellulosic materials (like Spain or Italy)
Ref: Marshall 1(1917), 340

Grille de pointage. Fr for Aiming Sight Grill

Grinding. See under Crushing and Grinding in Vol 3, p C564-R

Grisou. French for Firedamp

Grisou-dynamites are French Dynamites (Explosives containing NG) safe for use in coal mines. See Vol 1, p A467-L, under Antigrisou (Explosifs). They may be subdivided into two classes:
Grisou couche: Dynamites for use in coal seams; according to French Regulation, they should have a temp of expl below 1500°. Following are examples:
a) NG 12.0, Collod Cotton 0.5 & AN 87.5%. Temp of expl <1500°. Lead block expansion 245cc, Lead block crushing 8.5mm
b) NG 12.0, Collod Cotton 0.5, AN 82.5, K nitrate 50%, Temp of expl <1500°, Lead block expansion 235cc, Lead block crushing 7.5mm
c) NG 16, EGDN 4, Collod Cotton 0.5, AN 55, Cellulose 2.5, NaCl 22%. This expl is also called Chlorurée n° 1
Grisou roche: Dynamites for use in rock surrounding coal seams; they should have a temp of expl below 1900°
a) NG 29.4, Collod Cotton 0.6 & AN 70%. Temp of expl <1900°, Lead block expansion 295cc, Lead block crushing 18mm
b) NG 29.4, Collod Cotton 0.6, AN 65 & K nitrate 5%. Temp of expl <1900°, Lead block expansion 285cc, Lead block crushing 18mm
Note: The addition of 5% K nitrate instead of the same quantity of AN was found to increase the safety, probably because the K compds formed on expl dissociate at high temps thus absorbing part of the energy and lowering the overall temp
Grisou-dynamite chlorurées n° 1, n° 14 and n° 15 are in Vol 3, pp C450 & C451

Grisou-naphthalites. Fr coal-mining expls contg Nitronaphthalenes instead of NG. There are two classes:
a) *Grisou-naphthalite couche:* TNN 5.0 & AN 95%
b) *Grisou-naphthalite couche salpêtrée:* TNN 5, AN 90 & K nitrate 5%
c) *Grisou-naphthalite roche:* DNN 8.5 & AN 91.5%
d) *Grisou-naphthalite roche salpêtrée:* DNN 8.5,

AN 86.5 & K nitrate 5%
Grisou-tétrylite-couche. Fr permissible expl: AN 88, K nitrate 5 & Tetryl 7%
Refs: 1) Naoúm, NG (1928), 432, 433 2) Marshall **3**(1932), 121 3) Pepin Lehalleur (1935), 353 4) Davis (1943), 350

Grisounines. According to Gody (Ref), they were Favier type expls manufd by the Société Française des Poudres de Sûreté:
Grisounine roche: AN 91.5–92.0 & DNN 8.5%
Grisounine couche: AN 95.5 & TNN 4.5%
Note: The same expls are called by Gody on p 705 Grisoutines
Ref: Gody (1907), 594

Grisounites. Favier type expls, safe for use in gaseous coal mines. They are permissible Ammonium Nitrate expls which contn an aromatic nitrocompd in place of NG (See Antigrisous Favier under Antigrisou (Explosifs) (Vol **1**, p A467-L). These expls are identical with Grisou-naphthalites and Grisou-tétralite described above. They can be prepd by grinding the ingredients together in a wheel-milo (meule, in Fr), followed by sifting and packing in paraffined paper cartridges

Grisounite gomme. Accdg to Daniel (Ref 1, p 296), it was a mining expl contg AN 70 & NG 29.5, gelatinized with 0.5% Collod Cotton. Accdg to Naoúm (Ref 6, p 404), it consisted of AN 80, NG 19.6 & Collod Cotton 0.4%
Grisounite N_1c. Fr mining expl contg AN 87.4 & DNN 12.6% with detonvel 3635m/sec when compressed and 4100m/sec in powder form (Ref 4, p 493)
Grisounite N_8. Fr mining expl contg AN 15, Na nitrate 58 & TNN 27% with deton vel 3800 m/sec at d 1.38 or 3200m/sec at d 1.32 (Ref 4, p 493)
Grisounite-roche. Fr nonpermissible expl contg AN 92 & DNN 8%. It is listed here under Grisounines. Another Grisounite roche contd AN 70, NG 29.4 & Collod Cotton 0.6%. A similar expl contg AN 70, NG 29 & Collod Cotton 1% is called by Davis (Ref 8, p 351) Grisou-dynamite roche
Grisounite-tolite ou Grisoutolite. French expl contg TNT instead of TNN. For example, Grisoutolite-couche contd TNT 6.5, AN 88.5 & K nitrate 5.0% (Ref 6, p 433)
Refs: 1) Daniel (1902), 296 & 362 2) Gody (1907), 594 3) Marshall **1**(1917), 388, 389, 395 4) Marshall **2**(1917), 493 5) Colver (1918), 250, 275, 276, 494, 636 6) Naoúm, NG (1928), 384, 403, 303, 432, 433, 445 7) Pepin Lehalleur (1935), 343, 352–3 8) Davis (1943), 350–51 9) Clift & Fedoroff **2**(1943), G5 10 PATR **2700**, Vol **1**(1960), p A466-R

Grisoutina C; Grisoutina al 10% and **Grisoutina al 13.20%.** See Vol **3** of Encycl, p C451-R, under Italian Permissible Explosives

Grisoutines (Belgian & French). Blasting expls of which Gody (Ref 1) lists the following Belg compns:
Grisoutine I: NG 24.0, CC 1.0 & AN 75.0% (Ref 1, p 713)
Grisoutine II (d'Arendonck): NG 44.0, $Na_2SO_4.10H_2O$ 44.0 & woodflour 12.0% (Ref 1, p 714)

Gody also lists the following French compns:
Grisoutine d'Ablon dite B: NG 11.76, CC 0.34 & AN 88.0% (Ref 1, p 378)
Crisoutine A (Cugny): NG 4.85, CC 0.15, resin 4.55 & AN 90.45% (Ref 1, p 376)
Grisoutine B (Cugny): NG 5.875, CC 0.125 & AN 94.00% (Ref 1, p 376)
Grisoutine C (Cugny): NG 11.75, CC 0.25 & AN 88.0% (Ref 1, p 376)
Grisoutine D (Cugny): NG 19.50, CC 0.50 & AN 80.0% (Ref 1, p 376)
Grisoutine M (Société générale pour la fabrication des dynamites): AN 80 & Guhrdynamite of 75% NG 20% (Ref 1, p 359)
Grisoutine B (Paulilles): NG 11.76, CC 0.24 &AN 88.00% (Ref 1, p 705)
Grisoutine F (Paulilles): NG 19.6, CC 0.4 & AN 80.0% (Ref 1, p 705)
Grisoutine-gomme (Paulilles): NG 29.10, CC 0.9 & AN 70.0% (Ref 1, p 705)
Grisoutine No 2 (Paulilles): NG 35.0, cellulose 13.0 & Na nitrate 52.0% (Ref 1, p 705)
Grisoutine No 3 (Paulilles): NG 22.0, charcoal 12.0 & Na nitrate 66.0% (Ref 1, p 705)

Dautriche (Ref 2), as quoted from Ref 3, gave for French Grisoutines: NG 11.8 to 29.1, CC 0.2 to 0.9, AN 83.0 to 65.0 & Na nitrate 5%
Refs: 1) Gody (1907), 359, 376–78, 705 &

713–14 2) H. Dautriche, MP **16**, 52–54(1911–1912) 3) Clift & Fedoroff **2**(1943), p G5

Grisoutites (Belg). Permissible expls manufd at Matagne-la-Grande, such as NG 42 to 45, kieselguhr 11 to 12 & $MgSO_4.7H_2O$ 47 to 43% (Ref 1, p 358). Another compn is given on p 701 and in Ref 2: NG 44, cellulose 12 & $MgSO_4.7H_2O$ 44%

Refs: 1) Gody (1907), 358 & 701 2) Clift & Fedoroff **2**(1943), p G5

Grisou-tolite-couche. See Grisounite-tolite, under Grisounites

Grist is one of the names for size (fineness) of ground particles. Accdg to Marshall (Ref), the size of particles is of great importance in the expls industry. For instance, in the case of BkPdr, which contns no ingredient which is itself expl, it is of great importance to grind the ingredients very fine and to mix them intimately. In the case of mixts contg at least one substance which is an expl, coarser grinding and rougher mixing may suffice

Sometimes, as in the case of Permissible Explosives, it is undesirable to grind the ingredients too fine, as such grinding increases the density of the product and seems to increase the tendency to ignite firedamp when the expl is fired

Too fine grinding is also undesirable in expls contg a comparatively small amount of liquid expls, such as NG. It has been observed in such cases that they are much easier to detonate if the liquid is not absorbed completely by the non-expl ingredients (dope), but some free microscopic liquid particles are left imbedded between the particles of dope. Such droplets of liq expl serve as initiating centers. For this reason, in expls contg small amounts of NG, it is not advisable to use absorbents (such as wood pulp) which are ground too fine. Particle size affects greatly the rate of detonation of solid expls. In general, the smaller the size, the higher the rate of detonation

Ref: Marshall **2**(1917), 572

Grisúnaftalita. See under Spanish Permissible Explosives in Vol **3**, p C455-L

Grisutin. Russ expl consisting of NG gelatinized with Collodion Cotton 12–30 & AN 88–70%, with added absorbent such as sawdust (Ref 1). Accdg to J.E. Capell & A.B. Shilling of PicArsn (quoted from Ref 2) some cartridges 6 inches long and 1 to 1½ inches in diam, marked "Grisutin" were probably used for military purposes. Their compn was not detd at PicArsn

Refs: 1) Blinov, Vol **2**(1949) 2) B.T. Fedoroff et al, PATR **2145**(1955), p Rus 5

Grisutita capa and **Grisutita roca.** See under Spanish Permissible Explosives in Vol **3**, p C455-L

Grit. Fine stony or hard particles, such as sand, pieces of rock or metal, usually with rough surface

Ref: "The American College Dictionary", Random House, NY (1952), 533-R

Grit Influence on Initiation of Explosives by Impact and Friction.

High speed cameras reveal that initiation of expln in liquid and many of the solid expls. begins at "hot spots", generated either by the adiabatic compression of a gas pocket or by friction on a particle of grit, which creates the hot spot

According to Copp et al (Ref 1), sensitiveness to impact of PETN and other HE's is increased if grit of hardness greater than 4 on the Moh's scale is present. Bowden and Gurton (Refs 2, 3 & 4) claim that hardness over the range 2 to 7 is relatively of not so much importance as the mp of the grit. It has been claimed that only grits melting above 400°, or even 430°, increase the sensitiveness to impact of such expls as PETN, and that grits with mp of 500° and higher are necessary to cause increased sensitivity to LA, LSt and MF

Refs: 1) J.L. Copp et al, TransRoySoc(London), **A241**, 197–296(1948) & CA **42**, 7983(1948) 2) F.P. Bowden & O.A. Gurton, Nature **161**, 348 (1948) & CA **42**, 3963(1948) 3) F.P. Bowden & O.A. Gurton, Ibid, **162**, 654–5(1948) & CA **43**, 1982(1949) 4) F.P. Bowden & O.A. Gurton, ProcRoySoc **A198**, 337–49(1949) & CA **44**, 2241(1950)

Groggins, Philip H. American chemist, graduate of College of the City of New York (1912). Spent first 9 yrs with duPont at Gibbstown, NJ then moved from plant to plant for varied experiences. In 1935, he published the first edition of his monumental work "Unit Processes in Organic Synthesis". His first book was "Aniline and Its Derivatives"
Ref: Anon, C&EN **27**, 2423, 2457 (1949) & CA **43**, 7275 (1949)

Grommets. The term grommet as used in Ordnance means a ring formed of rope, wire, plastic material etc laid around rotating bands of projectiles shipped without packing. As rotating bands are made of softer metals than a projectile itself, they need extra protection which is achieved by using grommets

The simplest grommet consists of a piece of rope, looped at each end and wrapped once directly over the rotating band or in front of it, or in case of double-banded shell, between the bands. After this the hemp twine is drawn tightly thru the two end loops and secured by a "slipknot" (Knot designed to slide or pull free under strain)
Ref: US War Dept Tech Manual **TM 9-1901** (1950), pp 382-85

Gromoboy (Thunderer) (Russ). A powerful expl proposed in 1886 by I.M. Chel'tsov: AN 72.5 & Amm Picrate 27.5% for loading artillery projs and Naval mines
Refs: 1) Yaremenko & Svetlov (1957), p 5 2) Gorst (1957), 13

Groove Diameter of a rifled weapon is measured from the bottom of one groove to the bottom of that opposite or, in case an odd number of these are employed, calculated as equalizing the diam of the bore (See "Bore Diameter" in Vol **2** of Encycl, p B248-R) plus twice the depth of one groove, since in this instance these are so distributed that no one ever directly faces another
Ref: Hayes (1938), 194

Grottanelli Stability Test. A 500g sample of propInt as delivered (previously undried) is heated at 80° in a special glass reaction tube, while 0.05 liter of air, saturated with moisture, is passed thru it and then into bubbler tubes contg distd water. The acidity of the water in the bubbler tubes is measured periodically with a pH meter, without removin g the sample, and the heating continued for as many days as required to reach a pH of about 1.0. At this stage, the propInt is considered dangerous and the heating is discontinued

Pavlik (Ref 2) used a method which might be considered as a modification of both Grottanelli's and Hansen's methods. In this test, the pH was measured potentiometrically using a quinhydrone electrode. Déribéré (Ref 3) modified these methods by adding the water, used for washing the gases evolved during the stabilization test, directly to the container in which the propInt had been heated and determining the pH colorimetrically (instead of electrometrically) by a series of sensitive indicators (thymol blue for pH 2 to 3.3; bromophenol blue 2.8 to 4.6, bromocresol green 3.6 to 4.8; methyl orange 3.8 to 5.2; bromocresol purple 5.2 to 6.8 and bromothymo l blue 6.0 to 7.6)
Refs: 1) F. Grottanelli, MemorieRAccadItalia Vol **2**, No 6 (1931); MAF **14**, 503 (1935) & CA **26**, 2866 (1932) 2) A. Pavlik, Chim & Ind Special Number, 978-1005 (June 1933) & CA **28**, 326 (1934) 3) M. Déribéré, RevGénMat-Plastiques **10**, 260 (1934) & CA **28**, 6563 (1934) 4) Reilly (1938), 91 5) M. Tonegutti & E. Brandimarte, SS **35**, 124, 145, 169 (1940)

Ground Coke. The material prepd by finely grinding coke. Such material was used in primer compns such as MF 5, K chlorate 9, Sb_2S_3 3, Tetryl 2 & ground coke 1 part. It was also used in commercial expls, such as Black Dynamite, Dahmenite and Golovine Explosive, listed on p C292-L in Vol **3** of Encycl, under Coke

Ground Glass for Use in Primer Compositions. See under Glass

Grounding Against Lightning in Explosives and Ammunition Plants is described in *Refs:* 1) R. Rinkel, SS **6**, 141–45(1911)(Ueber den Blitzschutz von Sprengstoff-Fabriken) 2) A. Voigt, SS **6**, 204–05(1911)(Ueber Erdung von Pulverarbeitsmaschinen) 3) US Army Materiel Command, "Safety Manual", **AMCR 385–100**(April 1970), Chapter 9, "Lightning Protection", pp 8-1 to 8-36

Note: An interesting recent grounding method is employed in the Fábrica Naval de Explosivos at Azul, Argentina

Ground Signal Propellant. Its compn consisted of NC (13.25% N) 91.25, NG 5.5, DPhA 0.75, K nitrate 0.50, graphite 0.50 & Ba nitrate 1.50%. It was used in the form of a cross
Ref: Armament Engineering (1954), p 43

Growth of Explosion in Electrically Initiated RDX. It was studied both in powdered RDX initiated by a bursting bridgewire and in pressed RDX initiated by passing current thru a column of RDX-graphite mixt incorporated in the charge. In the former case, a reaction wave moves out from the bridgewire at a velocity characteristic of low-order deton, with a transition to high-order deton occurring at a reproducible induction distance which depends only weakly on the stored elec energy. Induction distances from 9.5mm to 17.5mm were observed, depending on geometry and loading d. Interactions of 2 pre-high order waves were investigated as well as the ability of such waves to cause deton in Comp B. In contrast, growth of expln in the pressed RDX appears to be governed, at least initially, by thermal mechanisms; delays of more than 1 msec have been observed between the discharge of the elec energy and substantial expln of the charge
Ref: G.M. Muller et al, JApplPhys **32**, 1065–75(1961) & CA **55**, 22825(1961)

Grüne prolosed in 1887 Dynamites contg as absorbents: charcoal mixed with kieselguhr, or kieselguhr alone previously heated to incandescence in the absence of air, and then cooled. It was claimed that such Dynamites do not exude NG even when brought in contact with water
Ref: Daniel (1902), 365

Grüneisen Equation of State is described in in Vol **4** of Encycl, p D278-L and *Mie-Grüneisen Equation of State* is on pp D289-L & R and p D290-L

Grüneisen Function, Effective is defined by Anderson et al (Ref) as "the ratio of the thermal pressure difference to the thermal energy density difference at a given volume". Values of the effective Grüneisen function are found to be in the range 1.4 to 2.2 and tend to decrease with decreasing volume and increasing temp
(Compare with Grüneisen Parameter, which is described under "Detonation, Equations of State": "Mie-Grüneisen Equation of State" in Vol **4**, p D289-L)
Ref: G.D. Anderson et al, "The Equation of State of 1060 Aluminum From Shock Wave Measurements", 4thONRSympDeton (1965), 213 (Abstract only)

GSX. Code name for 1,7-Dichloro-2,4,6-trinitro-2,4,6-triazaheptane described in Vol **5** of Encycl, p D1215-R

GTNB. Abbr for Glycol-di-trinitrobutyrate. See Ethyleneglycol-di-trinitrobutyrate in Vol **6**, p E255-R

Gu. Our abbr for Guanidine

Guaiacol and Derivatives
Guaiacol, Methylcatechol or *o-methoxyphenol* (Guaiacol or Brenzcatechinmonomethyläther in Ger), $HO.C_6H_4.OCH_3$; mw 124.13, prisms, sp gr 1.143 at 15/15°, mp 28.3°, bp 205°; sl sol in w; sol in org solvents. Prepd by heating catechol, alkali, and sodium methyl sulfate

at 175°. Forms an expl *picrate*, orn-red ndls, mp 80° (86°, 90°)
Refs: 1) Beil **6**, 768,(382), [776] & {4200}
2) E.V. Gorup-Besanez, Ann **147**, 248(1868)
3) CondChemDict (1950), p 328½ (1971), 427-R
4) Sax (1968), 801-L
Azido, $C_7H_7N_3O_2$, and *Diazido*, $C_7H_6N_6O_2$, derivs were not found in Beil

Mononitroguaiacols, $HO.C_6H_3(NO_2).OCH_3$; mw 169.13, N 8.28%. Four derivs are known:
3-Nitroguaiacol, yel ndls (subl), mp 62°; sol in w, steam distils. Prepd by nitration of guaiacol in HAc at RT
Refs: 1) Beil **6**, 788, (391), [789] & {4263}
2) H. Kaufmann & W. Franck, Ber **39**, 2725 (1906)

4-Nitroguaiacol, yel ndls (w), mp 104°. First prepd by saponification of the acetate, which had been prepd by nitration of guaiacolacetate in fuming nitric acid. *Sodium salt*, orn-red ndls, burns explosively in a flame
Refs: 1) Beil **6**, 788, (391), [790] & {4264}
2) R. Meldola, ProcChemSoc, No 167(1896), p 125

5-Nitroguaiacol, yel ndls (w), mp 99–100° (103–4°); sol in alc, eth & boiling w. Prepd by oxidation of 2-methoxybenzoquinone-1-4-oxime-4 with alkaline ferricyanide
Refs: 1) Beil **6**, 788, (391) & [790] 2) H. Rupe, Ber **30**, 2446(1897)

6-Nitroguaiacol, yel prisms or rhmb, mp 68.5–69.5°; sol in most org solvents except CCl_4, CS_2 & petr eth; insol in w. Prepd by saponification of the corresponding acetate
Refs: 1) Beil **6**, [789] 2) A.E. Oxford, JChemSoc **1926**, 2005

Dinitroguaiacols, $HO.C_6H_2(NO_2)_2.OCH_3$; mw 214.13, N 13.08%:
3,4-Dinitroguaiacol, yel prisms (tol), dec viol 200–08°; dif sol in organics. Prepd from 4-nitroguaiacol and nitric acid in HAc
Refs: 1) Beil **6**, (393) & [793] 2) F. Pollecoff & R. Robinson, JChemSoc **113**, 650(1918) & CA **12**, 2314(1918)

3,5-Dinitroguaiacol, yel plts or ndls (dil alc), mp 121–3°; sol in warm dil alc. Prepd from guaiacol in eth at 0° by adding nitrogen oxides from arsenic trioxide & nitric acid
Refs: 1) Beil **6**, 791, (394), [794] & {4273}
2) J. Herzig, Monatsh **3**, 825(1882)

3,6-Dinitroguaiacol, yel prisms (petr eth), mp 69–70°; sol in cold org solvents. Prepd from 3-nitrocatechol-2-methylether in cold HAc by adding nitric acid
Refs: 1) Beil **6**, [794] 2) A.E. Oxford, JChemSoc **1926**, 2008

4,6-Dinitroguaiacol, yel ndls (CS_2), mp 80°; sol in alc & EtAc; sl sol in benz, chlf & CS_2. Prepd by heating carbonic acid di(3,5-dinitro-2-methoxyphenylester) with aq-alc sodium carbonate
Refs: 1) Beil **6**, (394) 2) F. Pollecoff & R. Robinson, JChemSoc **113**, 649(1918) & CA **12**, 2314(1918)

5,6-Dinitroguaiacol, plates (CCl_4), mp 109.5–10°; sol in most org solvents except CCl_4, CS_2 & petr eth; dif sol in cold w. Prepd from 3-nitrocatechol-2-methyl ether in cold HAc by adding nitric acid
Refs: 1) Beil **6**, [793] 2) A.E. Oxford, JChemSoc **1926**, 2009

Trinitroguaiacols, $HO.C_6H(NO_2)_3.OCH_3$; mw 259.09, N 16.22%, OB to CO_2 –52.5%:
3,4,5-Trinitroguaiacol, yel crysts (w) or ndls (chlf-petr eth), dec 144–7°; sol in w, alc & eth. Prepd by boiling an HAc soln of the corresponding dimethyl ether with HBr. Its expl props were not investigated
Refs: 1) Beil **6**, [795] 2) M. Kohn & G. Löff, Monatsh **45**, 612(1925) & CA **20**, 1394 (1926)

3,4,6-Trinitroguaiacol, yel prisms (chlf), mp 129° (dec), expl weakly by heating under confinement. Prepd by cold nitration of 3,5-dinitrocatechol-2-methyl ether
Refs: 1) Beil **6**, (395) 2) F. Pollecoff & R. Robinson, JChemSoc **113**, 653(1918) & CA **12**, 2314(1918)

3,5,6-Trinitroguaiacol, monocl crysts, mp 113.5°, dec 131/, and yel plts, mp 108–9°, dec 145–75° depending on rate of heating; dif sol in cold w. Prepd by nitration of 3-nitrocatechol-2-methylether or of 3,6-dinitroguaiacol. Its expl props were not investigated
Refs: 1) Beil **6**, [795] 2) E.A. Oxford, JChemSoc **1926**, 2009 & CA **21**, 376(1927)

Tetranitroguaiacol, $HO.C_6(NO_2)_4.OCH_3$; not found in Beil

Guanazine. See Aminoguanazole in Vol 1 of Encycl, pp A209-R to A210-L

Guanazole. Same as 3,5-Diamino-asym-triazole, described in Vol **5**, p D1148-L & R. Its nitroso-, dinitroso-, nitro- and dinitro-derivatives are on pp D1148-R to D1149-R of Vol **5**
Addnl Refs: 1) P.G. Gordon, Compounds of High Nitrogen Content, Univ Microfilms, Publ No **7846**, 150pp (Diss Abstr **14**, 926–7)(1954) & CA **49**, 5452(1955) 2) ADL, Synth HE's, 3rd Rept (1953), pp 255, 257, 279 3) D.W. Kaiser & J.J. Roemer, USP 2648670–1 (1953) & CA **48**, 8268–9(1954) (Prepn of guanazoles from hydrazine or substituted hydrazine & NaN $NaN(CN)_2$) 4) R.W. Wiley & A.J. Hart, JOC **18**, 1368–71(1953) & CA **48**, 12092(1954) (Reaction of guanazole with diformyhydrazine) 5) D.W. Kaiser et al, JOC **18**, 1610–15(1953) & CA **48**, 13687(1954) (Dicyandiamide.V. Structures of guanazo- and pyroguanazoles, and reaction of dicyandiamide with 3-amino-5-substituted-4H-1,2,4-triazoles)

Guanazyl. The radical $\cdot C[:N.NH.C(:NH).NH_2].N:N.C_6H_5$ is termed guanazyl. Examples of compounds containing this radical are guanazylformic acid and guanazyl methane. The guanazyls do not form salts even with strong acids
Ref: E. Lieber & G.B.L. Smith, ChemRevs **25**, 248–50 (1939)

GUANIDINE AND DERIVATIVES

Guanidine (Gu) (Imino-urea)(Kohlensäure-diamid-imid, in Ger), $H_2N.C(:NH).NH_2$; mw 59.07, N 71.14%; colorless, very hygr crysts, mp about 50°, decomp at 160°; very sol in w. In addition to being very hygroscopic, Gu rapidly absorbs carbon dioxide from the air. In aq soln the base strength of Gu is comparable with that of NaOH. Gu was first prepd by Strecker in 1861 (Ref 2) by the oxidation of guanine (found in guano) with $KClO_4$ in the presence of HCl. Strecker also prepd these salts: carbonate, hydrochloride, nitrate, oxalate, and double salt of GuHCl with platinum chloride. He assigned the empirical formula $C_2H_5N_3$, but did not give a structural formula. A little later Erlenmeyer (Ref 3) prepd Gu salts by reacting an ammonium salt with an aq soln of cyanamide: $H_2N.CN + NH_4X = GuHX$. Currently Gu nitrate and hydrochloride are being made in the USA, with some carbonate being imported. The nitrate is prepd by reacting 1 equiv dicyandiamid (cyanoguanidine) with 2 equivs of Ammonium Nitrate in liq ammonia in an autoclave at 160° for 1 hr; this gives a 92% yield of 91% pure Gu Nitrate, which is adequate for the manuf of Nitroguanidine (NGu). The hydrochloride is prepd by heating ammonium chloride with dicyandiamid, dry. Other salts may be prepd by reacting a Gu salt with a salt of the "acid" desired. Gu may also be prepd by the ammonolysis of urea or thiourea:
$H_2N.C:X.NH_2 + NH_4Y = GuHY + H_2X$
Laboratory Preparation of Guanidine (American Cyanamid Co). Mix 100g of Gu carbonate with 400ml w and maintain below 25°. While stirring slowly, add a slurry of 43g of $Ca(OH)_2$ in 50ml w. Agitate for 15 mins and filter. Wash with small portions of w until filtrate plus washes approach 550ml. This soln will contain approx 10% free Gu, and should be stored under 25° to avoid decompn (See also Ref 5, p 13 for prepn of Gu from the sulfate). Four methods of prepn are listed in Refs 6 & 11
Properties of Guanidine. In aq soln at RT Gu will decomp in a few days to urea, ammonia, and carbon dioxide (faster at higher temp or in the presence of alkali). Gu forms salts, even with weak acids, the strong acid ones being stable to boiling w. Gu does not appear to be toxic, but one study (Ref 8) found this order of relative toxicity (in increasing order):

phosphate, carbonate, nitrate, chloride, and thiocyanate. Gu has large quantity usage in the synthesis of pharmaceuticals, dyes, cationic resins, and NGu. Some of the salts to be described next have found use in the expls industry, and may be prepd from the others. One study (Ref 10) found this order of thermal stability (decreasing): perchlorate, chloride, nitrate, and picrate

Other derivs which are expl or serve for prepn of expls are:
N-(β-Hydroxyethyl), N-nitroguanidine Nitrate (See Hydroxyethylguanidine under "H", and *Trinitrophenylnitroguanidine* (See Nitrophenylguanidine under "N")
Refs for Guanidine: 1) Beil **3**, 82, (39), [69] & {154} 2) A. Strecker, Ann **118**, 151–177(1861) 3) E. Erlenmeyer, Ann **146**, 258(1868) 4) Davis (1943), p 374 5) American Cyanamid Co, Inc, NY, Cyanamid's Chemical Digest, Vol IV, "The Chemistry of Guanidine" (1950) (Preparation, properties, methods of analysis, uses and toxicity of guanidine, its salts and derivatives, 456 refs) 6) CondChemDict (1961), 553-L 7) L.E. Craig & J.T. Minor, USP 3009949(1961) & CA **56**, 12748(1962) (Guanidine salts) (Prepn of Gu by heating Amm metaphosphate with urea at 250°; prepn of szlts by adding appropriate acid at end – nitrate & picrate) 8) A. Shansky, AmPerfumerCosmet **78**(12), 29–30(1963) & CA **60**, 11262(1964) (A presentation of the medical aspects of Gu salts) 9) Kirk & Othmer, Vol **10**(1966), pp 734–40 (E.H. Sheers, Guanidine and guanidine salts) 10) Y.P. Carignan & D.R. Satriana, JOC **32**(2), 285–9(1967) & CA **70**, 10960(1969) (Differential thermal analysis of nitramines, amine salts, and guanidine derivatives) 11) CondChemDict (1971), 428-L

Guanidine Carbonate (GuC), $(H_2N.C:NH.NH_2)_2H_2CO_3$; mw 204.19, N 41.18%; crysts, sp gr 1.251 at 30°, mp 241°; sol in g/100g: 45 in 20° w, 62 in 55° w, under 0.1 in 78° alc, under 0.05 in 50° acet, 60° benz, or 50° hex. GuC was prepd for the first time in 1861 by Strecker (See Ref 2 under Guanidine). It may be prepd by treating a boiling aq soln of dicyandiamid with carbon dioxide. Davis (Ref 2, p 385) describes the method of prepn, starting from NGu. Heat on a w bath a 2-liter flask fitted with a thermometer and a reflux condenser, and contg 208g Nitroguanidine (NGu), 300g ammonium carbonate and 1 liter w. When the temp reaches 65–70° and the nitrogen oxide gas starts to evolve, shake the flask to prevent excessive frothing which might carry over undissolved NGu. Gradually raise the temp until soln is complete. Remove the flask to a hot plate and boil for 2 hrs. Transfer the soln to an evaporating dish on a steam bath and heat to dryness, to decompose any residual Amm carbonate. Cool, then dissolve the residue in a small amount of cold w and filter to remove traces of melamine. Stir the filtrate with 2 vols of 95% alc to ppt the GuC, leaving impurities in soln. Filter, rinse with alc and dry. Evaporate the mother liquor to dryness, dissolve in a small amount of w, and re-ppt any remaining GuC. Total yield is 162g (90%)

GuC is an alkali of about the same strength as Na carbonate. It is relatively non-toxic (Ref 6). The heat of combstn at const vol and 18° is 2.566kcal/g (Ref 6)
Refs: 1) Beil **3**, 86, (40) & [72] 2) Davis (1943), 385 3) Cyanamid's Nitrogen Chemicals Digest, Vol IV, NY (1950) 4) L. Médard & M. Thomas, MP **37**, 129–38(1955) & CA **51**, 716(1957) (Heats of combustion of seven explosive or related substances) 5) CondChemDict (1961), 553-L 6) H. Bergner et al, ArchTierernaehr **19**(6), 421–32(1969) & CA **72**, 53306(1970) (Toxicity of new nonprotein nitrogen compounds in rats) 7) CondChemDict (1971), 428-L

Guanidine Chlorate (GuChl), $H_2N.C:NH.NH_2.HClO_3$; mw 143.54, N 29.28%, OB to CO_2 and Cl_2 –22.3%; colorless crysts, mp 148°(dec, Ref gives 98–100° for apparently pure material); sol in w. May be prepd by the double decompn of Gu Sulfate and Ba Chlorate, and evapn of the resultant soln over a w bath until crysts appear. As indicated in Ref 4, the thermal stability is not very high. This material is a mild expl which burns away when touched with a flame or a drop of sulfuric acid (concd). The same Ref indicates that its impact sensitivity is comparable to PA. It was proposed (Ref 3) as a component of expl mixtures with such fuels as C, S, Si, Al and an oxidizer like K nitrate

Refs: 1) Beil **3**, (40) 2) R.L. Datta & J.K. Choudhury, JACS **38**, 1083(1916) 3) G. Manuelli & L. Bernardini, BritP 155627(1917) & CA **15**, 1622(1921) 4) W. Markwald & F. Struwe, Ber **55**, 457(1922) & CA **16**, 2482(1922) (Preparation of Guanidine Chlorate and other guanidine salts) 5) Cyanamid's Nitrogen Chemicals Digest, Vol IV, NY (1950)

Guanidine Chromates (GuChr). Three chromate salts are known: the *mono* $(Gu)_2H_2CrO_4$; yel ndls, sp gr 2.301; the *di* $(Gu)_2H_2Cr_2O_7$; red-yel pl, sp gr 2.502, and the *tri* $(Gu)_2H_2Cr_3O_{10}$; red plates or bipyr, sp gr 2.595. There is also a *perchromate* $(Gu)_3H_3CrO_8.H_2O$, yel-brn prisms. The latter is prepd by adding 33% peroxide to freshly prepd "chromate", and demonstrates no expl props; by heating an aq soln of the perchromate one may obtn the monochromate. The other chromates, including mono, may be prepd by gentle heating of appropriate amounts of chromic anhydride and $Gu_2H_2CO_3$ in w. All are easily sol in w except the perchromate. Heating the dry materials causes decompn, and doing it confined causes explns – mild for the mono and violent (190°) for the di salt. The chromates have some use as corrosion inhibitors

Refs: 1) Beil **3**, 86 & [72] 2) K.A. Hofmann & K. Buchner, Ber **42**, 2773–6(1909) (mono & per) 3) R. Weinland & H. Staelin, ZeitAnorgChem **136**, 313(1925) (di & tri) 4) A. Swaryczewski, BullInternAcadPolanaise, Classe-SciMathNat **1934A**, 246–55 & CA **29**, 975(1935) (Crystallographic study of the mono-, di-, and trichromates of guanidine) 5) C.Ma, JACS **72**, 1333–5(1951) & CA **45**, 6111(1951) (Thermal decomposition of Guanidine Chromate and Dichromate) 6) A.F. Nagornyi et al, RussP 184836(1966) & CA **66**, 75708(1967) (Prepn of chromate by adding chromic anhydride to freshly prepd free guanidine)

Guanidine Fluorochromate, $H_2N.C:NH.NH_2.HCrO_3F$; mw 179.07; orn-yel scales, expl on heating; sol in w. May be prepd by dissolving 0.02 mole of chromic anhydride in a minimum of w, adding 5ml if 40% HF, then adding 0.02mole of $Gu_2H_2CO_3$, and evaporating over sulfuric acid. Darkens in daylight, etches glass even when dry, dec in damp air evolving HF, and apparently hydrolyzes in w to fluoride and chromate ions

Refs: 1) Beil **3**, [72] 2) R. Weinland & H. Staelin, ZeitAnorgChem **136**, 313(1925)

Guanidine Hydrochloride (Guanidiniumchlorid, in Ger) (Gu.HCl) ; $H_2N.C:NH.NH_2.HCl$; colorless ndls, sp gr 1.344 at 30°, mp 184°; sol in g/100g: 215 in 20° w, 320 in 55° w, 67 in 78° alc, insol in 50° acet, 60° benz, and 50° hex. Was first prepd by Strecker in 1861(Ref 2). It may be prepd by treating GuC with hydrochloric acid. Delitsch (Ref 3) made it from Gu thiocyanate, reacting with Cu sulfate to give Gu sulfate, which reacts with Ba hydroxide to give free Gu, and reacting with HCl. Gu-HCl is slightly toxic when taken internally; aq solns or wet Gu-HCl in contact with the skin are also toxic

It has been used as an exceptionally water-sol source of Gu for org syntheses (Ref 5)

Refs: 1) Beil **3**, 86 & [71] 2) A. Strecker, Ann **118**, 163(1861) 3) G. Delitsch, JPraktchem **9**, 5(1874) 4) Cyanamid's Nitrogen Chemicals Digest, Vol IV, NY (1950) 5) Cond-ChemDict (1961), 553-L & (1971), 428

Guanidine Nitrate (GuN), $H_2N.C:NH.NH_2.HNO_3$; mw 122.09, N 45.89%, OB to CO_2 –26.2%; colorless crysts, mp 214.2°, sp gr 1.436 at 30°; sol in g/100g: 15 in 20° w, 47 in 55° w, 200 in 100° w, 13 in 78° alc, sl in 50° acet, insol in 60° benz, and insol in 50° hex. May be prepd directly from calcium cyanamide by hydrolysis to cyanamide, which is heated to form dicyandiamide and then heated with two equivalents of AN at 160° to give GuN. Since this has the drawbacks of requiring 3mols of AN per mol of GuN made, and the expl hazard of carbonaceous impurities in the calcium cyanamide, the cyanamide or dicyanamide is preferably separated first before proceeding. Paden et al (Ref 5) describe a method starting with dicyandiamide and AN. Eventually liq ammonia was used as a diluent for the large amount of heat evolved. Older methods are given in Beil (Ref 1) and Davis (Ref 3, 378–9)

Laboratory Preparation. Davis (Ref 3, p 380) gives this method: On a 160° oil bath heat a 1-liter flask contg an intimate mixt of 210g dicyandiamide and 440g AN. After 2 hrs, cool to RT. Then place on a steam bath and extract

the contents with several portions of hot w, decanting each portion and combining all. Reheat to dissolve, and filter while hot to remove the insol ammeline and ammelide. Concentrate the soln to about 1 liter and cool. Decant the mother liquid and concentrate further to ¼ liter. Combine both crops, redissolve in the least amount of w; cool, filter, and dry. The product is suitable for laboratory prepn of NGu. GuN may be considered a very weak expl with Trauzl test value of only 10% TNT, deton vel of 3700m/s. It is very insensitive to impact, or even expl primers, failing to deton under the impact of a 10kg wt falling 3.1m (Ref 11). Heat of combustion is 1715cal/g at const vol and w (liq), and heat of formation variably given as 88–97kcal/m

GuN is used as an ingredient of some Blasting Expls and for the prepn of the expl NGu (Refs 15 & 19)

Refs: 1) Beil **3**, 86, (40) & [72] 2) G.B.L. Smith et al, IEC **23**, 1124(1931) (Quantitative study of the preparation of GuN and NGu, 30 refs) 3) Davis (1943), 374–80 4) K.G. Herring et al, IEC **38**, 1315–19(1946) (Improved prepn of GuN in 92–94% yield from "lime nitrogen", AN and urea) 5) J.H. Paden et al, IEC **39**, 958(1947) (Guanidine nitrate from dicyandiamide and AN by pressure reaction, 22 refs) 6) G.F. Wright, USP 2431301(1947) & CA **42**, 2616(1948) (Prepn of GuN by fusing Ca cyanamide and AN in the presence of sufficient urea to reduce the mp of the mixt to below 120°) 7) Honarary Advisory Council for Science and Ind Research, BritP 620030 (1949) & CA **43**, 5796(1949) (Prepn of GuN by fusing crude Ca cyanamide with AN and urea at a temp below 120°) 8) W.H. Hill, USP 2468067(1949) & CA **43**, 5416(1949) (Improved process for the prepn of GuN from Amm thiocyanate and AN. As the direct fusion of these two substances produces an expln, the new process calls for dissolving the first in liq ammonia, and then heating with AN at 120° in a closed vessel to produce GuN, ammonia and hydrogen sulfide) 9) Cyanamid's Nitrogen Chemical Digest, Vol IV, "Chemistry of Guanidine", American Cyanamid Co, NY(1950) 10) J.A. Grand & R.R. Miller, USP 2555333 (1951) & CA **45**, 7337(1951) (Use in solid fuel contg NGu 75, GuN 25, and Cu chromite ¼–½ parts; gives a large vol of reducing gas at moderate temp) 11) L. Médard, MP **33**, 113–23(1951) & CA **47**, 5683(1953) (Explosive properties of Urea Nitrate, Nitrourea, and GuN) 12) Belgrano (1952), p 132 (Use of GuN as an ingredient of Albite) 13) J. Barlot & S. Marsaule, MP ∝π, 349–64(1953) & CA **50**, 818 (1956) (Salts of aminoguanidine) (Eutectic: $GuNH_2.HNO_3$ 85 and GuN 15 parts, mp 126°) 14) G. Bourjol & G. Dumoulin, MP **39**, 97–104 (1957) & CA **52**, 19940(1958) (Continuous prepn of guanidine nitrate in an anhydrous medium) (Heating of AN and dicyandiamide) 15) CondChemDict (1961), 55e-L & R 16) D.Sh. Rozina et al, MetodyPoluchen iyaKhimReaktivoviPreparatov, GosKomSovMinSSSRPoKhim No **4–5**, 5–8(1962) & CA **61**, 571(1964) (Prepn of GuN from AN and Ca cyanamide) 17) N.V. Krivtsov et al, ZhNeorgKhim **10**(2), 454–7 (1965) & CA **62**, 12515(1965) (Enthalpy of formation of Guanidine Perchlorate, Nitrate, and Sulfate) 17a) Urbański, Vol **2**(1965), 466–69 17b) Sax (1968), 801-R 18) G.A. Lobanov & L.P. Karmanova, IzvVysshUchebZaved, KhimKhimTekhnol **14**(6), 865–7(1971) & CA **76**, 28438(1972) (Enthalpy of formation of some organic substances) 19) CondChemDict (1971), 428-R

Guanidine Nitroform (GuNf), $H_2N.C:NH.NH_2.HC(NO_2)_3$; mw 210.12, N 40.01%, OB to CO_2 –7.6%; yel ndls, sp gr 1.64,mp 128°(86.4–7.4° for a monohydrate); sl sol in cold w. Prepd by J.V.R. Kaufman of Picatinny Arsenal from a very dil aq Gu soln and Nitroform at RT. It may also be prepd from Gu_2-H_2CO_3 and Nitroform. The impact sensitivity with a 2kg wt on PicArsn App is 11" compared with 14" for TNT. Even though it is one of the most stable organic Nitroform salts, the termal stability is unsatisfactory. It explodes after 30 hrs in the 100° vacuum test. Deton vel is comparable with RDX. It does not react with dry aluminum at 90°

Refs: 1) Beil, not found 2) O.H. Johnson & F. Taylor Jr, NAVORD Rept **2125**(NOL) *(1951)* (Guanidine Nitroformate and Hydrazine Nitroformate as possible new HE's) 3) W.F. Sager & D.V. Sickman, NAVORD Rept **483**(1952) (Research and development in new chemical high explosives)

Guanidine Oxalate (GuOxl), $H_2N.C:NH.NH_2 .C_2 H_2 O_4$; mw 149.11, N 28.2%, OB to CO_2 −59.1%; colorless crysts from w as a hydrate, mp (dec above 200°); dif sol in cold w. First prepd by Strecker by treating Gu_2-H_2CO_3 with oxalic acid. Potentially a good flash-reducing agent

Refs: 1) Beil **3**, 86 & (40) 2) A. Strecker, Ann **118**, 162(1861) 3) M. Raffe & O. Balduzzi, GazzChimItal **47**, I, 70(1917)

Guanidine Perchlorate (GuPchl), $H_2N.C:NH.NH_2 .HClO_4$; mw 159.54, N 26.34%, OB to CO_2 and ½Cl −10.0%; colorless crysts, sp gr 1.1−1.67, mp 248°, bp (expl about 367°); sol in w (11.5%, w/w, at 0°), $HClO_4$ (36.7%, w/w, and forms a monoperchlorate solvate, mp 6±2°). May be prepd by mixing equimolecular amounts of Gu and $HClO_4$ or by heating dicyandiamide and Amm Perchlorate in an autoclave at 150−160°. GuPchl is a powerful expl, much more sensitive to impact than PA (50cm compared with 85cm with a 2kg wt on BurMines App). Deton vel is 6000m/s at sp gr 1.15, and 7150m/s at 1.67. The Pb Block Expansion (Trauzl Test) is 400cc for a 10g sample. In comparing the brisance with NGu and GuN by the Pb Plate Test, Stettbacher (Ref 9) found that these two only dented the plate, but GuPchl punctured it. GuPchl was proposed (Ref 2) as an ingredient of expl mixts, and was patented in Ger for use in expls (Ref 4)

Refs: 1) Beil **3**, (40) & [72] 2) C. Manuelli & L. Bernardini, BritP 155627(1917) & CA **15**, 1622(1921) 3) R.L. Datta & N.B. Chatterjee, JCS **11**, 1010(1919) 4) Chemische Fabrik Griesheim-Elektron, GerP 309297 & 309298 (1921) 5) W. Markwald & F. Struwe, Ber **55**, 457(1922) (Preparation and explosive properties of guanidine perchlorate) 6) Anon, Jahresber-Chem-TechnReichsanstalt **6**, 93(1927) 7) A. Stettbacher, Nitrocellulose **6**, 79(1935) 8) Ibid, **7**, 144(1936) 9) Davis (1943), p 366 10) K.V. Titova & V.Ya. Rosolovskii, ZhNeorgKhim **10**(1), 446−50(1965) & CA **62**, 11393 (1965) (Some physico-chemical properties of GuPchl) 11) N.V. Kritvtsov et al, ZhNeorgKhim **10**(1), 454−7(1965) & CA **62**, 12515 (1965) (Enthalpy of formation of Gu Perchlorate, Nitrate, and sulfate) (Gives the value −74kcal/m) 12) V.Ya. Rosolovskii et al, ZhNeorgKhim **12**(5), 1275−81(1967) & CA **67**, 85457(1967) (Perchloric acid as a solvent of organic perchlorates) 13) Ibid **13**(3), 681−6(1968) & CA **69**, 13538 (1968) (Energy of the addition of perchloric acid to amines and the heats of formation of amine perchlorates) (Give for the heat of formation of GuPchl-$HClO_4$ −97±1 kcal/m; for the heat of addition for GuPchl −45.9kcal/m- for the heat of addition of GuPchl-$HClO_4$ from GuPchl −14.7kcal/m) 14) R.N. Isaev et al, IzvVysshUchebZaved, KhimKhimTekhnol **13** (8), 1089−92(1970) & CA **73**, 132626(1970) (Effect of additives on the thermal stability of GuPchl and its mixts) (Stability decreased by 10% ferric oxide, and increased slightly by 10% ZnO) 15) J.J. Byrne, USP 3531338(1970) & CA **74**, 14697(1971) (Propellant contg GuPchl-LiPchl eutectics in homogeneous phase with polymeric binders) (GuPchl/LiPchl, mp: 70/30, 126.5°; 66.8/33.2, 151°; 57.5/42.5, 110°. These are much less sensitive to impact than LiPchl alone. The polymerization of the binder should not produce w, eg, acrylamide is acceptable)

Guanidine Picrate (GuPicr or GuP), $H_2N.C:NH.NH_2.HOC_6H_2(NO_2)_3$; mw 288.18, N 29.17%, OB to CO_2 −63.8%, sp gr about 1.5. GuPicr exists in two stereoisomeric forms:

Labile − lt yel ndls, sl sol in w (0.8 at 80°). May be prepd by reacting PA with free Gu

Stabile − deep yel plates, sl sol in w (0.574 at 80°); dif sol in alc & eth. May be prepd by the action of Gu on sol Picrates

Generally a mixt results on prepn, giving a mp range of 310−333° with decompn as low as 280°

GuPicr is an expl with deton vel about 6500m/s. It is less sensitive to impact and shock than AmmPicr and thus may be used as a filler for armor-piercing shells. Its thermal stability is satisfactory. Its use as an expl dates from about 1901 when it was patented in Ger for use in commercial expls. When mixed with sufficient K nitrate to give a zero oxygen balance, the resultant powdery mixture can absorb up to 1/3 its weight of NG, and becomes somewhat preferable to regular Dynamites (Ref 2). Olsen (Ref 3) patented GuPicr

as a filler for projectiles, alone, or blended with paraffin or TNT and compressed to no lower than sp gr 1.5. The following are some published formulations for commercial expls: GuPicr 40–30 and K nitrate 60–70, or GuPicr 75 and NG 25, or GuPicr 80–75 and $KClO_3$ 20–25%

Refs: 1) Beil **3**, 40 and **6**, 279, (135) & [265] 2) Colver (1918), p 341 3) F. Olsen, USP 1558565 (1925) & CA **20**, 112 (1926) 4) Davis (1943), p 168 5) M.I. Fauth, AnalChem **32**, 655–7 (1960) & CA **54**, 15934 (1960) (Differential thermal analysis and thermogravimetry of some salts of Gu and related compounds) (Compares thermal stability, deton temp and 5-kg impact sensitivity of Picrates and Styphnates of Hydrazine, aminoguanidine, N-methylguanidine, guanylurea, N-ethylguanidine and Gu)

Guanidine Sulfate (GuSulf), $(H_2N.C:NH.NH_2)_2\text{-}H_2SO_4\text{-}\frac{1}{2}H_2O$; mw 225.23, colorless crysts; sol in w; insol in alc & other org solvents. Was first prepd in 1861 by Strecker (Ref 2). May be prepd by treating dicyandiamide with 75% sulfuric acid at RT (Ref 3). Stettbacher (Ref 4) heated dicyandiamde with an excess of 50% sulfuric acid. More recently Mackey (Ref 5) fused 300g of urea with 1140g of Amm sulfamate, $NH_4SO_3NH_2$, at 225–45° for 30 mins, and then extracted with w to give 338g of GuS. Treating Gu carbonate with sulfuric acid is also effective

GuSulf may serve for the prepn of the free base by treating with Ba hydroxide in w, or with NaOH or $NaNH_2$ in liq ammonia. It has been used as a fireproofing agent, as an intermediate in the prepn of surfactants and pharmaceuticals and for prepn of resins

Refs: 1) Beil **3**, 86 2) S. Strecker, Ann **118**, 151–77 (1861) 3) P.A. Levene & J.K. Senior, JBiolChem **25**, 623–4 (1916) & CA **10**, 3067 (1916) 4) A. Stettbacher, Nitrocellulose **7**, 142 (1936) 5) J.S. Mackey, USP 2464247 (1949) & CA **43**, 4292 (1949) 5) Cyanamid's Nitrogen Chemicals Digest, Vol IV, "The Chemistry of Guanidine", American Cyanamid Co, NY (1950) 6) N.V. Krivtsov et al, ZhNeorgKhim **10**(1), 454–7 (1965) & CA **62**, 12515 (19659 (Enthalpy of formation of guanidine perchlorate, nitrate, and sulfate) (Give the value –287kcal/m)

Guanidine Tetraperoxoniobate, $(H_2N.C:NH.NH_2)_3\text{-}[Nb(O_2)_4]$; N 31.42%, OB to CO_2 and $\frac{1}{2}Nb_2O_5$ –37.9%; crysts, expl below 100° on rapid heating, dec 130° on slow heating; sol in w; insol in alc. Was prepd from $NbCl_5$, 10% H_2O_2, and $Gu_2\text{-}H_2CO_3$

Refs: 1) Beil, not found 2) R.N. Shchelokov et al, ZhNeorgKhim **16**(2), 402–5 (1971) & CA **74**, 93901 (1971) (Synthesis of some tetraperoxoniobates)

Guanidine Thiocyanate (GuTh), $H_2N.C:NH.NH_2\text{-}HNCS$; mw 118.17, N 47.4%, colorless deliq lflts, mp 118°; sol in w (134.93 g/100g at 15°), EtAc (20g/100g at 25°), acet & aq alc. Was first prepd by Delitsch in 1874 (Ref 2). Accdg to Davis (Ref 4) it may be prepd by heating Amm thiocyanate for 20 hrs at 170–90°, or until H_2S is no longer evolved. Presumably, the reaction involves the formation of thiourea, its conversion to cyanamide, and reaction of the latter with undecomposed thiocyanate. The storage instability of cyanamide precludes its direct use for this prepn. Werner & Bell (Ref 3) prepd GuTh in nearly theoretical yield by heating 1 mole of dicyandiamide with 2 moles of ammonium thiocyanate for 2 hrs at 160° to give 2 moles of GuTh. Ref 1 offers other methods

GuTh was formerly one of the most common salts of Gu, serving as the parent of other salts thru metathetical reactions. It also served as starting material for NGu by direct nitration with mixed acid. The NGu obtd proved to be unsuitable for use with NC because of the sulfurous impurities which reacted with the NC in storage. A strong aq soln of GuTh will dissolve cellulose (Ref 4)

Refs: 1) Beil **3**, 169, (70) & [121] 2) G. Delitsch, JPraktChem **9**, 1–6 (1874) 3) E.A. Werner & J. Bell, JCS **117**, 1133 (1920) 4) Davis (1943), pp 375, 379

Analytical Procedures and Tests for Guanidine and Its Salts. Gu, urea, their salts, covalent compounds, and polymers have the property of evolving ammonia when heated to 250°, which is not shared by other amines or amides. Thus, after a preliminary heating at 180° for several

minutes in a test tube of a small amount of sample, the tube is covered with filter paper treated with Nessler's Reagent and heating is continued to 250°; yel or brn coloration indicates the presence of any of these classes of material (Ref 6). Sullivan (Ref 1) describes a spot color test involving the reaction with 1,2-naphthoquinone-4-sulfonate. For quantitative analysis, the preferred method according to the article on Gu in Kirk & Othmer (Ref 19 under Gu) is potentiometric titration with $HClO_4$ in ethylene glycol methyl ether. Next best is the gravimetric determination involving precipitation with calcium picrate, most recently discussed by Fainer & Myers (Ref 5). A modification of the picrate method is discussed by Ghosh & Sarkar (Ref 9) who prefer to use magnesium bis-trinitrophenylamine, $Mg[N(TNB)_2]_2$, as the precipitant. The min detectable amount of GuN is ½% in w, and, with precautions, no interference from AN, urea, melamine or dicyandiamide is encountered

Refs: 1) A. Vezarick, ZAngewChem **15**, 70 (1902) (Method of determination of Gu and its derivatives) 1a) M.X. Sullivan, ProcSocExptl-BioMed **33**, 106–8(1935) & CA **30**, 8074(1936) 2) M.X. Sullivan & W.C. Hess, JACS **58**, 47 (1936) & CA **30**, 1746(1936) 3) A.M. Soldate & R.M. Noyes, AnalChem **19**, 442–4(1947) & CA **41**, 6105(1947) (X-ray diffraction patterns for the identification of crystalline constituents of explosives) (Includes GuPicr) 4) Cyanamid's Nitrogen Chemicals Digest, Vol IV, "The Chemistry of Guanidine", pp 37–8(1950) 5) P. Fainer & J.L. Myers, AnalChem **24**, 515–17(1952) & CA **46**, 6041 (1952) (Estimation of guanidine with Ca Picrate) 6) F. Feigl, "Spot Tests in Organic Analysis", 5th ed, Elsevier, Amsterdam (1956), pp 291–2 7) B.B. Coldwell, Analyst **84**, 665–7(1959) & CA **54**, 17887(1960) (The application of ultraviolet light and diphenylamine to spot tests for explosives) (GuN & NGu among others) 8) A.S. Jones & T.W. Thompson, JChromatog **10**(2), 248(1963) & CA **59**, 15582(1963) (Detection of guanidine compounds on paper chromatograms) (Gu, GuHCl, GuN are detected with ninhydrin-NaOH) 9) Y.P. Carignan, JOC **32**(2), 285–89(1967) & CA **70**, 10960(1969) (Differential thermal analysis of Gu derivs) 10) P. Ghosh & J.M. Sarkar, Technology **5**(1), 7–9(1968) & CA **70**, 31716 (1969) (Gravimetric estimation of guanidine by Hexanitrodiphenylamine) 10a) Ibid, **5**(3), 229–30(1968) & CA **70**, 109571(1969) (Properties of Guanidine Dipicrylamine complex) (Explodes at 254°) 11) J. Bartos, Talanta **16**(4), 551–3(1969) & CA **71**, 9479(1969) (Colorimetry of alkylamines, guanidines, and quaternary ammonium salts by the Janovsky Reaction) (Free Gu from its salt with AgO in the presence of nitromethane and TNB; colorimetrically measure the red compd formed)

NITROGUANIDINE (NGu)(Picrite)(Gudol in Ger), $H_2N.C:NH.NH.NO_2$; mw 104.07, N 53.84%, OB to CO_2 −30.8%; sp gr 1.81, mp 232° [if the temp be raised at a moderate rate, values between 220 and 250° have been obtained (Ref 12)]. NGu was first prepd in 1877 by Jousselin [CR **85**, 548(1947) & **88**, 874(1879)] by dissolving dry GuN in fuming nitric acid and passing nitrous oxide thru the soln. By drowning the soln in w, he obtd a ppt which he called "Nitrosoguanidine", but which was later proved to be NGu by J. Thiele [Ann **270**, 1(1892)]. Thiele found that it is easier to prep NGu by treating GuN with sulfuric acid (See below). Davis & Elderfield [JACS **55**, 731(1933)] found that phosphoric acid is also an effective dehydrating agent

NGu exists in at least 2 cryst modifications, an *alpha* and a *beta*. The alpha form may be prepd by dissolving GuN in concd sulfuric acid and drowning in w. It crystallizes from hot w as long, thin, flexible, lustrous ndls which are tough and pulverize with difficulty. It is the form most commonly used in the expl industry. The beta form may be prepd (along with a small amount of alpha) by nitrating a mixture of GuSulfate and Amm sulfate. It crystallizes from hot w in fern-like clusters of small, thin, elongated plates. The beta may be converted into the alpha by dissoln in concd sulfuric acid and drowning in w

Laboratory preparation of NGu. Davis (Ref 6d, p 381) gives this procedure: Immerse a 1-liter beaker contg ½ liter of concd sulfuric acid into cracked ice and stir until the contents are at 10° or less. In small portions add 400g of dry GuN with stirring to keep the temp under 11°. When all of the GuN has dissolved, drown the milky liquid in 3 liters of cracked ice and w. Let stand until completely pptd, then filter thru a Büchner funnel and rinse to remove

excess acid. To purify, dissolve in about 4 liters of boiling w and let stand overnight. Filter and dry to get a 90% yield

Plant preparation of NGu:

1st method: By dissolving GuN in concd sulfuric acid in the manner described in Laboratory Procedure. However, correspondingly larger amounts are used, depending on the size of the equipment

2nd method: By fusing equimolecular quantities of urea and AN:

$$H_2N.CO.NH_2 + NH_4NO_3 \rightarrow H_2N.C(:NH).NH.NO_2 + 2H_2O,$$

and then recrystallizing the product from boiling w (yield about 92%)

3rd method: By heating a soln of equimolecular quantities of cyanamide and AN in an autoclave at 160° and 200 lbs pressure:

$$H_2N.CN + NH_4NO_3 \rightarrow H_2N.C(:NH).NH.NO_2 + H_2O,$$

and then recrystallizing the product from boiling w (yield about 88%)

For many years (before and for sometime after WWI), NGu, as well as many other derivs of Gu, was prepd from Gu thiocyanate, one of the cheapest and easiest to prepare of the Gu salts. The thiocyanate used to be the principal industrial product of Gu. However, NGu prepd from thiocyanate by direct nitration with mixed acids always contained traces of sulfur compds. When such NGu was used with NC in smokeless proplnt, the sulfur compds attacked the NC and thus lowered the stability of the proplnt. This was one reason why NGu powder did not come into earlier use (Ref 6d, p 375)

Both forms of NGu have the same mp (about 232°) and are alike in most of their chemical and physical props. However, they differ slightly in solubility in w except at 25° and 100°, where their solubility curves cross, giving 0.42–0.44g per 100ml at 25° and 8.25g at 100°. Solubility between 25 and 100° is slightly lower for the α- than for the β-form, while at 0° it is about 0.12 for both forms. Both forms are sl sol in alc and nearly insol in eth. Solubility in 1N KOH is 1.2 at 25°

Solubility in aqueous H_2SO_4 (Ref 6d, p 383):

$\%H_2SO_4$	°C	g, in 100cc
0	0	0.12
	25	0.42
15	0	0.3
	25	0.55
20	0	0.45
	25	1.05
25	0	0.75
	25	1.8
30	0	1.3
	25	2.9
35	0	2.0
	25	5.2
40	0	3.4
	25	8.0
45	0	5.8
	25	10.9

Explosive Properties of NGu:

Booster Sensitivity Test: Min wt of Tetryl pellet at sp gr 1.41 to produce 50% detons is 100g, with 0.67 wax spacer (Refs 22a & 29b)

Brisance: Plate dent test, about 95% of TNT at sp gr 1.5. Lead block crushing – 40g of NGu placed on a block 67mm in diam produced a shortening of 7mm compared with 10.5mm for PA (Ref 6d, p 391 and Refs 22a & 29b)

Brisance by Sand Test: 36.0g (TNT 48.0g) or 73.5% of TNT (Refs 22a & 29b)

Covolume: 1.08 (Ref 6d, p 339)

Detonation Velocity: 5360m/s at sp gr 1.0 and 7650 at sp gr 1.5 (Refs 22a & 29b)

Explosion Temp: dec at 275° (5 sec) (Refs 22a & 29b)

Force: 9660 (Ref 6d, p 391)

Friction Pendulum Test – unaffected by fiber or steel shoe (Refs 22a & 29b)

Gas Volume: 1077cc/g (Refs 22a & 29b)

Heat of Combustion: 1995cal/g (Refs 22a & 29b)

Heat of Explosion: 721cal/g (Refs 22a & 29b)

Heat of Formation: 227cal/g (Refs 22a & 29b)

Heat Test at 100°: Loss in wt 0.48 in 1st 48 hrs, 0.09% in 2nd 48 hrs and no expln in 100 hrs (Refs 22a & 29b)

Hygroscopicity: non-hygroscopic at 30° & 90% RH (Refs 22a & 29b)

Impact Sensitivity: BofM App, 2kg wt, 47cm, or more sensitive than either TNT or PA (Refs 22a & 29ba); PicArsn App, 1 lb wt 26 inches (Refs 22a & 29b)

International Test at 75°: Loss in wt 0.04% in 48 hrs (Refs 22a & 29b)

Power: Ballistic mortar, 104% TNT; Trauzl test, 101% TNT (Refs 22a & 29b)
Pressure of Explosion (max): 4078kg/sq cm at sp gr (loading) 0.03 (Ref 6d, p 389)
Rifle Bullet Impact – unaffected (Refs 22a & 29b)
Sensitivity to Initiation – min chge LA 0.20g & Tetryl 0.10g (Refs 22a & 29b)
Storage: (dry)
Temp of Explosion: about 2098° as compared to 2820° for TNT. Patart (Ref 3, p 153) gives the temp for NGu as low as 907°, but this appears to be erroneous
Thermal Stability: satisfactory (Refs 22a & 29b)
Vacuum Stability at 100°: 0.37cc gas evolved by 1 g in 48 hrs (Refs 22a & 29b)
Volatility: none (Refs 22a & 29b)

The above data indicate that NGu is a HE resembling TNT and PA in its props, although giving a considerably lower sand test value
Uses: During WWI, NGu (50) was used by the Germans, in admixtures with AN (30) and paraffin was (20%), for filling trench mortar shells (Ref 6d, p 391)

Due to the low temp of expln of NGu, the possibility of its use as a temp reducing agent was investigated as early as 1901 by Vieille (Ref 2, p 195). This famous French expl expert found that, if 10 to 15% of NGu was incorporated in NC, the resulting proplnt was practically flashless and less erosive than those with expls of comparable force (See also Ref 6d, p 388). Investigations with NGu were carried out later in France by Patart (Ref 3) and Muraour & Aunis (Ref 6); in the US by Davis, Ashdown, Elderfield (Ref 4); Davis & Abrams (Ref 5); Smith (Ref 5a); and in Germany by chemists of the Dynamit AG and others. The use of NGu in proplnts was, however, rather limited because the commercial product attacked NC in storage. This seemed to be due to the presence of sulfur compds in the NGu, derived from the Gu thiocyanate, which served, usually, as a primary material for the prepn of NGu (See Note, under prepn of NGu). When the method of prepn was changed and the sulfur compds were absent, it was possible to prep more stable proplnts

German Gen U. Gallwitz, killed in action on the Russian front during WWII, was one under whose direction the cool, flashless, non-erosive proplnts contg NGu and DEGN were developed in Germany before and during WWII. Proplnt contg NGu was called *Gudol Pulver* (abbr "G" Pulver) in Germany (Ref 8, p 80, Ref 9 and cross refs listed under GUDOL PULVER). The first successful Gudol Pulver was prepd in Germany in 1937 by Dynamit AG. This proplnt gave muzzle flashlessness in light field howitzers without the addition of suppressing agents (flash-reducing agents) such as K sulfate, and the quantity of smoke was small. In the case of guns of larger caliber, the charges required comparatively small additions of flash-reducing agents to render them flashless. Addition of NGu not only suppresses the muzzle flash, but also eliminates the "breech flash", which is especially noticeable in modern guns (used in armored vehicles) equipped with muzzle brakes and automatic and semi-automatic closures. A most particular advantage of Gudol proplnt is the increase of the barrel life

Following are examples of some Gudol proplnts:
1) NC (12% N) 44.00, DEGN 18.85, NGu 20.00, DNT 3.50, α-Nitronaphthalene 2.00, diphenylurethane 1.50, ethylphenylurethane 1.50, K nitrate 4.00, hydrocellulose 4.00, akardite 0.40, MgO 0.15 & graphite 0.10%. This proplnt has a heat of combstn (calorific value) of 720cal/g (compared with 950 and 820 cal for some NG proplnts) and is suitable for various guns. When used in anti-aircraft guns, this proplnt increased the barrel life of a gun from 1700 firings (as experienced with NG proplnts with a calorific value of 950 cal) to about 15000 firings (Ref 8, p 88)
2) NC (12% N) 42.45, DEGDN 18.20, NGu 25.00, diphenylurethane 4.50, ethylphenylurethane 4.50, K sulfate 5.00, MgO 0.25 & graphite 0.10%. This proplnt had a calorific value of 730cal/g and was used in most of the Naval guns (Ref 8, p 91). NGu was also used as an ingredient of proplnts by other countries, including the US

Following is an example of an American NGu proplnt, as given in Spec MIL-P-668A (Feb 1955): NC (13.3 to 13.4% N) 18.0 to 22, NG 18–20, NGu 52.7–56.7, ethyl centralite 5.40–6.60 & cryolite 0.20–0.40%. It should be noted that flashless colloided proplnts contg NGu produce a considerable amount of particulate gray smoke (more than other proplnts used

in the US) smelling of ammonia (Ref 6d, p 387) [See also "Cool (or Cooled) Propellants" in Vol 3 of Encycl, pp C509-R & C510-L]

References on Nitroguanidine: 1) Beil **3**, 126, (59) & [100] 2) P. Vieille, MP **11**, 195(1901) 3) G. Patart, MP **13**, 153, 159(1905–06) 4) T.L. Davis et al, JACS **47**, 1063(1935) 5) T.L. Davis & A.J.J. Abrams, ProcAmerAcad-ArtsSci **61**, 437(1926) & CA **21**, 1968(1927) (Transformations of Nitroguanidine) 5a) G.B.L. Smith et al, IEC **23**, 1127–29(1931) (Preparation of Nitroguanidine) 6) H. Muraour & G. Aunis, MP **25**, 91(1932–33) 6a) A. Stettbacher, Nitrocellulose **7**, 141–5(1936) & CA **30**, 8165(1936) (Methods of prepn of NGu from dicyandiamide and from GuSulfate are described. In the latter, 100g of GuSulfate were nitrated with 360ml of mixed acid contg 98.2g nitric. The brisance of NGu as determined by the Lead Plate Test proved to be lower than that for the corresponding amount of GuPchl) 6b) T. Urbański & J. Skrzynecki, RocznikiChem **16**, 353–8(1936) & CA **31**, 2502(1937) (Thermal analysis of mixtured containing AN, GuN and NGu) (The following eutectics were obtd: AN 80, NGu 20% at 131.5°; GuN 59, NGu 41% at 166.5°; AN 60, GuN 22.5, NGu 17.5% at 113.2°) 6c) R.C. Elderfield, "Explosives from Hydroxy and Amino Compounds", OSRD No **158**(1942) PB Rept No 31094(1942) and OSRD No **907** (1942) PB Rept No 31085(1942) 6d) Davis (1943), 380–91 7) Bebie (1943), p 110 7a) W.H. Rinkenbach, PATR **1336**(1943) (Thermochemical and physical tests of NGu proplnts) 8) O.W. Stickland, "General Summary of German Explosive Plants", US Office of Tech Service, PB Rept No **925**(1945) 9) U. Gallwitz, "German Powder Development from 1918 to 1942", PB Rept No **47059**(1945) 9a) Amer Cyanamid Co, BritP 571527(1945) & CA **41**, 1700(1947) (Crystalline NGu in an extremely fine state of subdivision) & BritP 572231(1945) & CA **41**, 6896(1947) (Crystalline NGu) 10) J.H. Paden et al, IEC **39**, 952–58(1947) 10a) E.J. Pritchard & G.F. Wright, CanJRes **25F**, 257–63 (1947) & CA **42**, 369(1948) (Production of NGu with high bulk density) 10b) G.H. Foster & E.F. Williams USP 2445478(1948) & CA **42**, 7986(1948) [Rapid crystn of NGu from aq soln in the presence of a compd having an amino or subst amino group (such as ethylenediamine) produces small, very stable crysts (2½–20 micron) suitable for incorporation in propellants] 10c) C.M. Mason & G. von Elbe, "The Physics and Chemistry of Explosives Phenomena", Ordn Project TA 3–5001, USBurMines (June 1948), p 4(Sol of NGu in dimethylformamide is 10.6% at 30° and 13.9% at 40°) 10d) M. Dutour, MP **31**, 73–80(1949) & CA **46**, 11686(1952) (Explosive properties of NGu) [Sensitivity to initiation as a function of density; compressibility; coefficient of practical utilization (CUP) alone and mixed with Al; vel of deton as a function of density; impact, gap, and combustion tests] 11) Cyanamid's Nitrogen Chemical Digest, Vol IV, "The Chemistry of Guanidine", Am Cyanamide Co, NY (1950) 11a) G. Bourjol, MP **32**, 11–25(1950) & CA **47**, 3243(1953) (Preparation of NGu by dehydration of GuN with concd sulfuric acid): (GuN plus enough 94–95% sulfuric to give 85–88% acid at end of reaction, at RT but all right to reach 35–40° at the end) 12) W.C. McCrone, AnalChem **23**, 205–6(1951) (Crystalline structure of NGu as determined at atmospheric pressure – orthorhombic system. NGu sublimes before melting to give a few separate distinct crysts. Melting occurs at 246–47° with considerable decompn. If the melting process is stopped before completion, the unsublimed portion which was almost completely melted, crystallizes as fine needle aggregates in a terrace-like pattern, characteristic of compounds that melt with decompn) 13) J.N. Pring, USP 2557463(1951) & CA **45**, 9863(1951) (Proplnt compn of NGu 55, NC 19, NG 18.7 & DEtDPhUrea 7.3% may be prepd by solventless or solvent process to have a specific surface of 9000–22000sq cm/cc and a calorific value of 650–825cal/g) 14) A.F. McKay et al, CanJChem **29**, 746–58(1951) & CA **46**, 2501 (1952) (Structures of NGu and its derivs, 18 refs) 15) Ibid, 391–7(1951) & CA **46**, 7095 (1952) [Prepn of substituted NGu's and the cyclization of 1-(3-Nitroxybutyl)-3-nitroguanidine] [An amine or diamine was used to displace the MeN(NO)- radical from MeN(NO.C:NH-$NHNO_2$ giving eg, 1,2-bis(3-Nitroguanidino)-ethane, mp 248°(dec); 1,3-bis(3-Nitroguanidino)-propane, mp 234°(dec); 1,3-bis(3-Nitroguanidino)butane, mp 230°(dec); 1,4-bis(3-Nitroguanidino)butane, dec over 265°; 1-(3-Nitroxy; butyl)-3-nitroguanidine, mp 125°] 16) A.F. McKay, ChemRev **51**, 301–46(1952) & CA **47**,

4844(1953) (Nitroguanidines) 17) V. Ostojic, Technika(Belgrade) **12**, 770–4(1957) & CA **52**, 14171(1958) (Crystallization of NGu for obtg finely crystallized powder suitable for expls and use of protective colloids, preferably aqueous starch or gelatin) 18) W. Schemuth, GerP 1010888 & 1013556(1957) & CA **54**, 7936, 9774 (1960) (NGu with a large specific surface area and sulfuric acid-free NGu) 19) M. Thoma & H. Hagn, GerP 1004090 & 1005426(1957) & CA **54**, 18961(1960) (NGu with a high specific surface area and treatment of the NGu-sulfuric acid reaction mixture) 20) R.A. Henry & J. Cohen, USP 2946820(1960) & CA **55**, 393(1961) (High bulk-density NGu and recrystn of NGu from aq soln contg a little HAc and a little of a deriv of NGu such as hydrazone, hydrazide, amide, amine, sulfonamide, sulfate) 20a) CondChemDict (1961), 805-R (NGu) 21) L.D. Sadwin, Science **143**(3611), 1164, 1169(1964) & CA **60**, 14188(1964) (Explosive welding with NGu) 22) Kirk & Othmer **8**(1965), p 625–6(NGu) 22a) Anon, "Properties of Explosives of Military Interest", **AMCP 706–177**(1967), pp 239–42 23) Y.P. Carignan & D.R. Satriana, JOC **32**(2), 285–9(1967) & CA **70**, 10960(1969) (Differential thermal analysis of NGu's, amine salts, and Gu derivs) 23a) Poudreries Réunies de Belgique, SA, BelgP 717436(1968) & CA **71**, 51832(1969) (NGu recovery from triple-base expls; crush under w to about 1 mm, extract the Nitroglycerol with methylene chloride; extract the NGu from the residue with w) 24) J. Savitt, Wrst States Sect, CombustnInst(Pap) **1968**, WCS/CI-68-31, 10pp & CA **73**, 16945 (1970) (Detonation sensitivity of very low density pressings of NGu) (At 0.45–1.00g/ml in a 2-inch diam cardboard confinement, sensitivity to a No 8 cap decreases normally as sp gr increases; at 0.16–0.45g/ml, the sensitivity increases as sp gr increases) 25) E.I. Isaev et al, TrKonfAnalKhimNevodnRastvorovIkhFizKhimSvoistv, 1st **1968**, No 1, 65–8 & CA **72**, 96451(1970) (Effect of nonaqueous solvents on acid-base properties of NGu) (Acidic in dimethylformamide, pyridine and acet; basic in HAc and formic acid) 26) E. Ripper, Explosivst **17**(7), 145–51(1969) & CA **72**, 48454(1970) (α- and β-Nitroguanidine) (Reinvestigation of both forms by IR, UV, Nuclear Magnetic Resonance (NMR), Differential Thermal Analysis (DTA), thermogravimetric & X-ray showed marked differences in crystal habit and behavior to heat, the α- dec at loe er temp) 27) D. Price & A.R. Clairmont, Jr, 12thSympCombstn (1968), (Pub 1969), 761–70 & CA **75**, 8070(1971) (Explosive behavior of NGu) (Crit diam of high bulk-density is about 3X that of low bulk-density, but the relative difference in shock sensitivity is not that great) 28) W. Kemula et al, BullAçad-PolonSci, SerSciChim **18**(8), 455–61(1970) & CA **74**, 7187(1971) (N-nitroderivatives. Equilibriums of Nitrourea and NGu in aq soln) (NGu and its tautomeric acid form are in equilibrium at pH 13.6) 29) E. Ripper, ChimInd, Génie-Chim **103**(14), 1763–65(1970) & CA **75**, 8055 (1971) (α- and β-NGu) (Review of available data) 29a) CondChemDict (1971), 623-R 29b) **AMCP 706–177**(1971), Same pp as in Ref 22a 30) G.A. Lobanov & L.P. Karmanova, IzvVysshUchebZaved,KhimKhimTekhnol **14**(6), 865–7(1971) & CA **76**, 28438(1972) (Enthalpy of formation of some organic substances) (NGu–22.4±0.4 for 1–3mm crysts, and –23.9±0.6 for 0.2–0.8mm crysts)

Nitroguanidine Explosives

As has been mentioned under "Nitroguanidine", many expl mistures were patented in which NGu was used in small or large quantities. Its principal use, however, is in smokeless proplnts, due to its property of eliminating flash. Not only is it used in colloided NC proplnts, but Hale and Olsen have tried it as a component of smokeless proplnts contg no NC

NGu was also proposed for use in expl compns, together with other ingredients, such as fuels (C, S, Al, Si) and oxidizing agents (K nitrate, K chlorate, K perchlorate)

Following are additional refs of NGu expls: A) C. Manuelli & L. Bernardini, USP 1409963 (1922) & CA **16**, 1868(1922) (Use of NGu and of various NGu & Gu salts, such as the chlorate, nitrate and perchlorate, in various expl mixts. Other ingredients may be fuels and oxidizers) B) G.C. Hale & F. Olsen, USP 1547808 & 1550960(1925) and CA **19**, 3021(1925) & **20**, 112(1926) (A propellant which gives uniform results, is formed of NGu 41, PETN 21, NH_4ClO_4 32 & triphenylphosphate 6%. NGu may also be

incorporated in NC proplnts. For this, NGu is previously treated with a solvent, such as tolylmethylketone, benzaldehyde, diacetin or urethane, which also acts as a colloiding agent for the NC)
C) T.L. Davis, USP 1754417(1930) & CA **24**, 2886(1930) (An expl is formed, comprising NGu and an aliphatic Nitramine, such as methyl-, ethyl-, dimethyl- or methylbutylnitramine. NC may also be included)
D) S. Gordon & E. Whitworth, BritP 616898 (1949) & VA **43**, 4856(1949) (Flashless proplnts, which cause very little erosion, are prepd by incorporating in the usual smokeless proplnts large amounts of NGu and dibutylphthalate, eg, NGu 55±1, DBuPh 10±0.5, NC (colloidable) 20±1, NG 13±1 & diethyldiphenylurea 2±0.3%)
E) A.T. Tyre, USP 2470082(1949) & CA **43**, 5190(1949) (Permissible expls may be comprised of the following compounds: NGu, GuN, Nitrodicyandiamidine, or dicyandiamidine nitrate mixed with NaH_2PO_2, $Ca(H_2PO_2)_2$, the hypophosphites amounting to 5–35% of the total. Such mixts, when ignited, produce gas but no flame)
F) K.U. Holker, USP 2904420(1959) & CA **54**, 867(1960 (Self-sustained, non-detonating, gas-producing compositions) [One formula is: GuN 32, AN 33.8, K nitrate 3.8, dolomite 24.8, Amm dichromate (sensitizer) 3.7, and Cu oxalate (sensitizer) 2%; NGu may also be used instead of GuN]
G) A. Schloetzer, GerP 1102023(1961) & CA **57**, 10097(1962) (Polyvinyl nitrate (14.3% N) was combined with an equal amt of NGu to give a workable proplnt with a Q_e of 834cal/kg & a gas vol of 1246cc)H) R.L. Trask, S. Sage & I.G. Nadel, USP 3086896(1963) & CA **59**, 377(1963) (Proplnt contg NGu 50–65, NC (12.6% N) 16–23, NG 16–21, dialkylphthalate plasticizer 4.5 & stabilizer 1.5%)
I) E. Richter & R. Meyer, GerP 1696381(1970) & CA **72**, 123585(1970) (100p NG is added with agitation to 20p 25% alc NC (13.15% N), aged for 6 hrs to produce a clear cuttable, gelatinous mass which is kneaded for 4 hrs with an addnl 110p NC and 120p NGu, with addn of 40p acet and 30p alc to produce a dry proplnt of good stability & quality)
J) H. Shinozaki, D. Kameyama & F. Azuma, JapP 7027359(1970) & CA **74**, 89335(1971) (An expl powd contg NGu 9.7, Amm Nitrate 77.7, starch 11.6 and w 1%, is applied to polyethylene sheet overlaid stainless steel sheet and detonated from one end with a percussion cap)
K) H. Schleuter & F. Hermann, GerP 1944844 (1971) & CA **74**, 143986(1971) (Pressed expls of low percussion sensitivity contd NGu and 5–12% eutectic mixts of $2,4,6(O_2N)_3C_6H_2$-$NMeNO_2$ and its N-Et analog. NGu contg 10% eutectic had 8400m/sec deton vel, compared with 7600 for pure NGu)

Nitroguanidine Nitrate, $H_2N.C:NH.NH.NO_2$-HNO_3; mw167.09, OB to CO_2 +4.8%; rhomb prisms, mp 147° (dec). May be prepd by dissolving NGu in hot, concd nitric acid and allowing to cool. Loses the nitric acid in contact with air. The salt is an expl detonable by impact, but not by heat

Refs: 1) Beil **3**, 127 & [101] 2) J. Thiele, Ann **270**, 20(1892) 3) T.L. Davis et al, JACS **47**, 1065(1925) 4) Davis (1943), p 381

Nitroguanidine Perchlorate and Diperchlorate, $H_2N.C:NH.NH.NO_2$-$HClO_4$ and -$2HClO_4$; mw 204.53 and 304.99, N 27.45 and 18.36%, OB to CO_2 and ½ or 1 Cl_2 +11.7 and +26.2%. The monoperchlorate forms colorless, hygr crysts which dec to NGu and $HClO_4$ at 120°; may be prepd by dissoln of NGu in anhydrous $HClO_4$; sol in $HClO_4$ to 48.1%, wt/wt. The energy of addition in forming this salt is −21.1kcal/mole; the heat of formation is −51.5kcal/mole. The diperchlorate may be prepd by dissolving the mono- in $HClO_4$. It melts at 70° and explodes at 93°

Refs: 1) Beil, not found 2) V.Ya. Rosolovskii & K.V. Titova, ZhNeorgKhim **11**(12), 2819–20 (1966) & CA **66**, 121654(1967) (Nitroguanidinium Perchlorate) 3) Ibid **12**(5), 1275–81(1967) & CA **67**, 85457(1967) (Perchloric acid as a solvent of organic perchlorates) 4) Ibid **13**(3), 681–6(1968) & CA **69**, 13538(1968) (Energy of the addition of perchloric acid to amines and the heats of formation of amine perchlorates)

Nitroguanidine Silver Salt, $H_2N.C:NH.NAgNO_2$; mw 210.95, N 26.54%, OB to CO_2 and $\frac{1}{2}Ag_2O$ −15.2%; wh ndls (hot ammonia soln), expl or deflgr on heating; insol in w; sol in acid or ammonia. May be prepd by treating a soln of Ag nitrate in warm w with NGu in the presence of $Ba(OH)_2$. It gives an alkaline reaction
Refs: 1) Beil **3**, 127 2) J. Thiele, Ann **270**, 19(1892)

Nitroguanidine and Derivatives, Analytical Procedures.

Qualitative Tests for NGu. The presence of NGu may be detected by one of the following tests (Ref 1, p 384):

A) To about 0.01g of sample dissolved in 4ml of cold w, add 2 drops of saturated ferrous ammonium sulfate soln, followed by 1 ml of 6N NaOH. Allow to stand for 2 mins and filter. If NGu is present, the filtrate will have a fuchsin color, which usually fades after standing ½ hour

B) Mix about 0.1g of sample, 5ml of w and 1 ml of 50% acetic acid in a test tube and warm at 40–50° until the soln is complete. Add about 1g of Zn dust and place the test tube in a beaker of cold w for 15 mins. Filter and add to the filtrate about 1cc of 5% Cu sulfate soln. If NGu is present, the soln becomes extremely blue. Now, if this soln is heated to boiling, it is observed that gas evolves, then the soln becomes turbid and finally a ppt of metallic Cu is deposited. If instead of Cu sulfate soln, 1ml of Ag acetate soln is added and the liq boiled, a ppt of metallic Ag is formed

Note: Silver acetate soln is prepd by shaking with warming 2g CH_3COOAg crysts in soln of 2ml glac AcOH in 100ml of w, filtering and allowing to cool

Colorimetric tests for NGu are found in Ref 20, p C418-R, Vol **3** of Encycl under Tests for Propellants

Quantitative Analysis of NGu

Accdg to StdMethodsChemAnalysis (Ref 10a, p 1336), the assay of NGu is made by the conventional nitrometer method (described on p 755 of Vol 1 of StdMethodsChemAnalysis) on a sample that has been dried at 98 to 102°C (Nitrometer method is described in Vol **1** of this Encycl, pp A373-R to A377-R with illustration on p A374-L and also in this Vol)

It may alternately be detd by the method described on p 1394 of Ref 10a under "Nitroguanidine by Buffered Titanous Chloride Reduction"
Picatinny Arsenal Method. The following titration method developed at PicArsn as a culmination of many years work involving the Ti(III) titrant for nitro and nitrate species in general. It was published in the open literature in Ref 8

Prepare, standardize, and store 0.2N titanous chloride accdg to MIL-STD-286, Method 601.1. Similarly deal with 0.15N ferric Amm sulfate, same MIL, Method 603.1. Prep a buffer soln by adding 7ml of 30% NaOH per 25ml of a soln 1/1, wt/vol of Na acetate trihydrate in w. Prep a 20% Amm thiocyanate indicator soln. For the titration flask use a ½-liter flat-bottom boiling flask fitted with a S/T 24/40 female joint and a sealed in gas inlet tube. Into a 1-liter volumetric flask weigh a sample contg about 2.7g NGu. Add ¾-liter of hot, distd w with swirling to dissolve the sample. Let cool to RT, then dilute to 1 liter with more distd w and mix. Transfer 50ml of this soln to the titration flask already contg 50ml of the titanous soln and 25ml of the buffer soln deaerated with CO_2 (the flow of which is not interrupted until the analysis is complete). Magnetically stir the flask contents for 3 mins. Add 25ml of 1/1 HCl soln and 5ml of the indicator soln. Titrate the excess titanous ion with the standard ferric soln. Using the same reagents and procedure run a blank. Then, %NGu = 1.7345 N(A−B)/W, where (A−B) represents the corrected volume of ferric titrant, N is the actual normality of the latter, and W is the weight of the sample, in g, represented by the aliquot taken from the volumetric. The results are at least as accurate, and probably more reliable than those obtd by the nitrometer method described in the JAN-N-494 Spec mentioned in the previous section. The main advantage of this reverse titration method is that the NGu has much less opportunity to decompose in the alkaline condition imposed by the buffer before being reduced by the Ti(III)

Note: In the method describing detn of TNT in Dynamites, pp D1646-L to D1648-R of Vol **5** of Encycl is given an illustration of "Apparatus for storing and using titanous chloride solution". There is also described on p D1646-L the method of prepn of std 0.2N $TiCl_3$ soln from ti-

tanium hydride. The procedure was developed by Mr N. Liszt of Picatinny Arsenal

Another, lengthier, method involving transnitration of salicylic acid by the NGu is described in Ref 4. It may not retain otherwise good accuracy on very finely divided NGu. Still other methods are described in the Refs, and Ref 7 compares some of the better known ones

References for the Analysis of Nitroguanidine:
1) Davis (1943), pp 380–91 2) D.L. Kouba et al, AnalChem **20**, 948–9 (1948) & CA **43**, 1568 (1949) (Determination of nitro nitrogen in NGu and Cyclotrimethylenetrinitramine) (Titanous chloride method) 3) G. Bourjol & M. Teindas, MP **31**, 51–71 (1949) & CA **46**, 11684 (1952) (Analytical methods applicable to NGu manufg control, including the urea type chemicals and their salts which are generally encountered) 4) H. Stalcup & R.W. Williams, AnalChem **27**, 543–6 (1955) & CA **49**, 8736 (1955) (Volumetric detn of NC and NGu by transnitration of salicylic acid) 5) J.E. DeVries et al, Anal Chem **27**, 1814–15 (1955) & CA **50**, 2369 (1956) [Titrations of weak acids and bases related to NGu (Study of acid-base props of NGu). Titration with $NaOCH_3$ in dimethylformamide, or with $HClO_4$ in trifluoroacetic acid gives good results] 6) P. Aubertein & H. Pascal, MP **40** 113–25 (1958) & CA **54**, 25825 (1960) [Chemical detn of some expls and expl mixts) (NGu is hydrolyzed in concd sulfuric acid, followed by volumetric detn of nitric acid by ferrous sulfate)] 7) M.I. Fauth & H. Stalcup, AnalChem **30**, 1670–72 (1958) & CA **53**, 2929 (1959) [Evaluation of six methods for detn of nitrogen in NGu. (Nitro N methods tested are: modified nitrometer – greatest precision of the six; acetate-buffered titanous chloride; transnitration with sulfuric acid; and Bowman-Scott). Total N methods are: modified Kjeldahl and micro-Dumas – better of these two. The titanous chloride and transnitration methods are satisfactory.]
8) M. Roth & R.F. Wegman, AnalChem **30**, 2036–38 (1958) & CA **54**, 2092 (1960) (Detn of NGu by reduction with buffered titanous chloride)
9) B.B. Coldwell, Analyst **84**, 665–7 (1959) & CA **54**, 17887 (1960) (The application of ultraviolet light and diphenylamine to spot tests for expls NGu and GuN among others)
10) L. Marvillet & J. Tranchant, MP **42**, 271–84 (1960) & CA **55**, 12855 (1961) (Titanometric detn of nitrogen. Application to propellants and their constituents) 10a) StandardMethods-ChemAnalysis, Vol **2B** (1963), 1393–94 (NG in proplnts) 11) V.M. Aksenenko et al, Zavodsk-Lab **31** (10), 1191 (1965) & CA **64**, 22 (1966) [(Potentiometric detn of NGu) (Acet solvent with Et_4- or Bu_4NOH as titrant)] 12) G.F. Atknin et al, ZavodskLab **32** (7), 802–4 (1966) & CA **65**, 12854 (1966) (Polarographic detn of NGu) 13) H. Opel & W. Ardelt, ZChem **7** (11), 439–40 (1967) & CA **68**, 46066 (1968) [(Analysis of cyanamide derivatives. III. Determination of NGu) (Method is polarographic dilution titration with borax buffer and Na sulfite – compares well with titanous chloride method)]

Nitroguanidine, Requirements and Tests of. US Military Specification MIL-N-494A of March 1963 (superseding JAN-N-494 of 10 Sept, 1947), with Amendment 3 of 31 Jan 1964 (superseding Amendments 1 and 2) and with Engineering Order from Picatinny Arsenal No EOPA-45940-S of 7 Dec 1966

1. SCOPE

1.1 Scope. This specification covers Nitroguanidine for use in the manuf of proplnts

1.2 Classification. NGu shall be of the following classes and types as specified (see 6.1 and 6.2):

Type I – Minimum purity 98%
Type II – Minimum purity 99%
Class 1 – Average particle diam 6.0 microns maximum & 3.4 microns minimum (See Table II)
Class 2 – Average particle diam 3.3 microns maximum (See Table II)

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein

STANDARDS

Military

MIL-STD-105 – Sampling Procedures and Tables for inspection by Attributes
MIL-STD-109 – Quality Assurance Terms and Definitions
MIL-STD-129 – Marking for Shipment and Storage
MIL-STD-286 – Propellants, Solid. Sampling, Examination and Testing

MIL-STD-1233 – Procedure for Determining Particle Size Distribution and Packing Density of Powdered Material
MIL-STD-1235 – Single and Multilevel Continuous Sampling Procedures and Tests for Inspection by Attributes
2.2 Other Publications
See Spec, pp 1 & 2
3. REQUIREMENTS
3.1 Material. NGu, as received, shall be a white, free flowing, crystalline powder, when tested as specified in 4.3
3.2 Properties. NGu shall be in accordance with the chemical properties specified in Table I, when tested in accordance with the applicable subparagraphs specified therein (See 6.4)

Table I

Properties	Type I, %		Type II, %	
	Min	Max	Min	Max
Purity (assay)	98.0	–	99.0	–
Ash content	–	0.30	–	0.30
pH value	4.5	7.0	4.5	7.0
Acidity (as sulfuric acid)	–	0.06	–	0.06
Total volatiles	–	0.25	–	0.25
Sulfates (as sodium sulfate)	–	0.20	–	0.20
Water insol impurities	–	0.20	–	0.20

3.3 Average particle size. NGu shall comply with the average particle size specified in Table II, when tested in accordance with 4.3.8

Table II

Average Particle Size – Microns

Class	Minimum	Maximum
1	3.4	6.0
2	–	3.3

3.4 Workmanship. The NGu and all containers shall be free from dirt, oil, grease, wood chips and other foreign material
4. QUALITY ASSURANCE PROVISIONS (p 2 of Spec)
4.1 **General quality assurance provisions** (p 2)
4.1.1 *Contractor quality assurance system* (pp 2–3)
4.1.2 *Submission of product* (p 3)
4.1.3 *Government verification* (p 3)
4.1.3.1 *Surveillance* (pp 3–4)
4.1.3.2 *Product inspection* (p 4)
4.2 **Inspection provisions** (p 4)
4.2.1 *Lot formation* (p 4)

A lot shall consist of one or more batches of NGu, produced by one manufacturer in accordance with the same spec or its revision, under one continuous set of operating conditions. Each batch shall consist of that quantity of NGu that has been subjected to the same mixing process intended to make the final product homogeneous
4.2.2 *Examination.* Sampling plans and procedures for the following classification of defects shall be in accordance eith Standard MIL-STD-105. Continuous sampling plans, in accordance with Standard MIL-STD-1235 may be used if approved by the procuring activity
4.2.3 *Sampling by lot.* A random sample of containers shall be selected from each lot in accordance with the following table:

Table III

Lot Size	Sample Size
1 batch	8 containers
2 batches	12 containers
3 or more batches	16 containers

When lots are composed of 8 containers or less each container shall be sampled
4.2.3.1 *Preparation of composite.* Equal primary samples in sufficient quantity to total 16 oz shall be removed from each container. The individual primary samples shall then be combined in order to form a homogeneous composite sample of 16 oz and subjected to the tests specified in 4.3. If the composite sample fails to comply with any of the requirements specified, the lot shall be rejected
4.3 The following test methods and procedures shall be used:
4.3.1 *Determination of purity.* The purity shall be detd by either the nitrometer method or the alternative chromous chloride method
4.3.1.1 *Nitrometer method.* The nitrometer method specified in Standard MIL-STD-286, method 209.3 shall be used as the standard method for purity except that a 3g sample shall be crushed so that it will pass thru a US Standard No 100 sieve. A 1 g sample shall be dried in air at 46°C overnight and then placed in an oven for 1 hr at 100°. The sample shall then be transferred to the nitrometer. The percent nitrogen

shall then be converted to percent NGu by the following formula:

$$\text{Percent NGu} = \frac{7.430R}{W}$$

where: R = reading of measuring tube
W = sample weight
7.430 = correction factor

Note: Detailed description of nitrometer method with illustration is given in Vol 1 of Encycl, pp A373-R to A376-R

4.3.1.2 *Chromous Chloride Method*

4.3.1.2.1 *Chromous Chkoride (0.2N) Standardization.* A 110g sample of hydrated chromous chloride shall be dissolved in about 500ml of distd w contd in a 2-liter volumetric flask. Fifty ml of concd HCl shall br added and diluted to the mark with distd w. After mixing, the soln shall be poured into a 4-liter bottle contg an excess of amalgamated zinc. The mixt shall be allowed to stand overnight and maintained under this atmosphere at all times. A bright blue color indicates complete reduction of chromic ions (green) to chromous ions (blue). The chromous chloride solution shall be transferred to an automatic buret with a 2-liter reservoir

4.3.1.2.2 *Ferric Alum (0.15N) Standardization.* The 0.15N Ferric Alum soln shall be prepd as specified in Standard MIL-STD-286, method 603.1

4.3.1.2.3 *Procedure.* An accurately weighed portion of approx 0.1g of sample shall be transferred to a 500-nl titration flask. Twenty-five ml of distd w shall be added and shaken and heated until the sample is dissolved completely. The titration flask shall be placed with a magnetic stirrer and the contents shall be stirred for at least 10 mins. The air shall be swept out with a stream of carbon dioxide and the carbon dioxide atm shall be maintained thruout the detn. Exactly 50ml of 0.2N chromous chloride soln shall be added to the 500ml titration flask. The contents shall be stirred for 5 mins and 25ml of 15 percent HCl shall be added. Ten drops of 2% aq phenosafranin indicator shall be added and the excess chromous chloride shall be titrated with 0.15N ferric alum soln. The end point is a sharp color change from green to blood red. A blank detn using the same procedure and reagents but omitting the sample shall be conducted. The percentage of NGu shall be calcd according to the following equation:

$$\text{Percent NGu} = \frac{1.735N(V_1 - V_2)}{W}$$

where: V_1 = ml of ferric alum used to titrate the total chromous chloride (blank)
V_2 = ml of ferric alum used to titrate the excess chromous chloride
W = weight of sample in g used in the analysis
N = normality of ferric alum soln

Note: Illustration of "Apparatus for Storing and Using Titanous Chloride Solution" is on p D1647 of Vol 5. On p D1646-L is stated that Mr N. Liszt developed a method of prepn of $TiCl_3$ from titanous hydride

4.3.2 *Determination of ash content.* The ash content shall be determined as specified in Standard MIL-STD-286, method 106.1

4.3.3 *Determination of pH value.* A portion of 5.0g of the sample, weighed to the nearest mg shall be transferred to an 8 oz bottle which has been previously heated to 80°C. A 200ml portion of freshly boiled distd w, which has been allowed to cool to 80°, shall be added. The bottle and contents shall be immersed in a constant temp w bath, maintd at 80°±2°. The bottle shall be removed for shaking at 3-min intervals. As soon as the soln is complete, the bottle shall be removed and rapidly cooled to RT in cold running tap water. The pH of the soln shall be detd using a glass electrode pH meter. This soln shall be retained for the "Determination of Acidity" (See 4.3.4). Prior to the pH detn, the meter shall be calibrated using standard buffer solns, at pH 4.0 and pH 8.0

4.3.4 *Determination of acidity.* The soln retained from the detn of the pH value shall be titrated with a 0.05N NaOH soln to a reading of 7.6 on the pH meter. A blank detn shall be conducted on an equal volume of distd w and the volume of NaOH used for the specimen shall be corrected. The percent acid present in the soln shall be calcd to sulfuric acid as follows:

$$\text{Percent sulfuric acid} = \frac{4.9(A-B)N}{W}$$

where: A = ml of NaOH soln used for titrating specimen
B = ml of NaOH soln used for titrating blank
N = normality of NaOH soln
W = weight of specimen in g

4.3.5 *Determination of total volatiles.* A portion of the sample, approx 5.0g, weighed to the

nearest 0.1mg, shall be transferred to a tared Al dish(90mm in diam and 60mm deep) having a tightly fitting cover. The uncovered dish contg the specimen shall be heated for 2 hrs in an oven maintd at 100°±2°C. At the end of this period the dish shall be covered, transferred to a desiccator until cool, and then weighed. The procedure of heating, cooling in a desiccator, and weighing shall be repeated at hourly intervals until 2 successive weighings do not differ by more than 0.2mg, or the specimen increases in weight. The percent total volatiles shall be calcd as follows:

$$\text{Percent total volatiles} = \frac{(A-B)\,100}{A}$$

where: A = weight of specimen in g prior to heating
B = weight of specimen in g after heating

4.3.6 *Determination of sulfates.* A portion of the sample, approx 5.00g, weighed to the nearest 0.1mg, shall be dissolved in 200ml of hot distd w contd in a 400-ml beaker. If necessary the soln shall be filtered thru a No 42 Whatman filter paper, or equal. The soln shall be made acid by the addn of 1 ml of concd HCl, which shall be followed by the addn of 1 ml of 10% Ba chloride soln. The soln shall be boiled for approx 5 mins and then allowed to stand, with occasional gentle stirrings, until cool. The soln shall then be allowed to stand for at least 10 hrs. At the end of this period, the soln shall be heated to boiling and filtered thru a previously ignited and tared Gooch crucible equipped with a fine asbestos mat. The ppt shall be washed with small portions of warm distd w, sucking each portion thru before adding the next. The last few washing shall be tested with 1% Ag nitrate soln to see that all chlorides have been removed. When free of chlorides the Gooch crucible shall be transferred to an oven maintd at 100°±2°C. followed by 1 hour in a muffle furnace, maintd at 600°C. The crucible shall then be transferred to a desiccator until cool, and weighed. The percent sulfate present in the specimen shall be calcd to Na sulfate as follows:

$$\text{Percent Na sulfate} = \frac{60.86A}{W}$$

where: A = weight of residue in g
W = weight of specimen in g

4.3.7 *Determination of water insoluble impurities.* A portion of the sample of approx 10.0g, weighed to the nearest 0.1g shall be dissolved in 400ml of boiling distd w. The soln shall be filtered thru a tared Gooch crucible equipped with a fine asbestos mat. The residue shall be washed with a total of 100ml of boiling w, in 20ml portions, letting each portion go thru the crucible before adding the next. The crucible shall be transferred to an oven, maintd at 105°±5° for 2 hrs, cooled in a desiccator and weighed. The gain in weight shall be calcd as percent water insol impurities as follows:

$$\text{Percent water insoluble impurities} = \frac{(A-B)\,100}{W}$$

where: A = weight of crucible & residue in g
B = weight of crucible in g
W = weight of specimen in g

4.3.8 *Average particle size.* The average particle size shall be detd in accordance with MIL-STD-1233, method 100

Nitrosoguanidine (NsoGu), $H_2N.C{:}NH.NHNO$; mw 88.07, N 63.63%, OB to CO_2 −54.5%; yel ndls, sp gr 1.52, explodes 161°; sl sol in cold w; sol in hot w; insol in alc & eth. May be prepd by reducing NGu with Zn powder in dil sulfuric acid, or by treating a soln of Gu carbonate in dil sulfuric with Na nitrite. NsoGu explodes on contact with concd sulfuric acid. It is sensitive to shock, friction, and burns, especially, with a very cool flame. The FI value is 93% PA, the Pb block value 60% PA, heat of combstn (w liq) 2.22kcal/g and heat of expln 99cal/g. Slowly dec in w, but may be commercially transported only with at least 10% w present. Manufd in the 1950's by Redel Inc, Anaheim Calif
Refs: 1) Beil **3**, 124, (59) & [99] 2) T.L. Davis & A.J. Abrams, ProcAmerAcadArtsSci **61**, 444–57(1926) & CA **21**, 1968(1927) 3) F. Olsen & F. Seavy, USP 2060522(1937) & CA **31**, 542(1937) (Expl priming mixts contg NsoGu) 4) Bebie (1943), p 111 5) Davis (1943), pp 391–3 6) A. Okáč & J. Gruber, ChemListy **45**, 49–51(1951) & CA **45**, 6541(1951) [(Formation of NsoGu salts and their analytical evaluation). (The Ag, Cu, and Ni salts were prepd in hopes of their being useful for analysis of these metals as the system is very similar to glyoxime; the results were not favorable. The

Ag salt was found to be expl)] 7) W.A. Gey & A.L. Bennet, JChemPhys **23**, 1979–80(1955) & CA **50**, 2174(1956) [Sensitivity of expls to pure shocks. (NsoGu and other expls were tested for their sensitivity to the impact of various power shock waves)] 8) Redel Inc (Anaheim, Calif), DataSheetNo 5601(1956) 9) CondChemDict (1961), 808.L and (1971), 625-R

Other Guanidine and Guanyl Derivatives

The remaining derivatives contg the guanidine, [:N.C(NH).N:] and guanyl [:N.C(NH).] structural features have been somewhat arbitrarily alphabetized as (in order): derivatives of guanidine, guanidinium derivatives, guanidino derivatives, and guanyl derivatives. Alkyl and nitroxyalkyl guanidines, following the pattern established earlier in this work, may be found under the alkyl, and hydroxyalkyl names, respectively, unless the particular alkyl radical was not previously entered

1-(1,1-Dimethyl-2-nitroxyethyl)-3-nitroguanidine, $O_2NO.CH_2.C(CH_3)_2.NH.C(:NH).NH.NO_2$; mw 221.17, N 31.67%, OB to CO_2 −76.0%; crysts, mp 112–14° (resolidifies, and melts again at 175–7°). Prepd by treating the unnitrated compd with mixed acid at 0°

Refs: 1) Beil, not found 2) L. Fishbein & J.A. Gallaghan, JACS **76**, 3217–29(1954) & CA **49**, 8990(1955)

1-Methyleneamino-3-nitroguanidine $O_2N.NH.C(:NH).NH.N:CH_2$; mw 131.09, N 53.44%, OB −55.0%, crysts, mp 169°. Prepd as a deriv of formaldehyde by reacting the latter with aminonitroguanidine

Refs: 1) Beil, not found 2) W.F. Whitmore et al, JACS **57**, 706–7(1935) & CA **29**, 3319(1935)

1-Nitro-3-(2,2,2-trifluoroethyl)guanidine, $O_2N.NH.C(:NH).NH.CH_2.CF_3$; mw 186.09, N 30.11%, OB −43.0% (to CO_2, CF_2O, HF, H_2O, N_2); crysts, mp 147–8°. Prepd by reacting trifluoroethylamine with S-methyl isothiourea in boiling w

Refs: 1) Beil, not found 2) V. Milani et al, JACS **77**, 2903(1955) & CA **50**, 3224(1956)

Nonafluorodiaminomethylguanidine, $F_2N.C(:NF).NF.C(NF_2)_2F$; mw 265.04, N 26.42%, FB +7.2% 'FB is analogous with OB, being applied hereafter to fluorinated compds wherein the F is not already on C; it is calculated in the same manner, the factor 1900/mw being used instead of 1600/mw); liq, bp ca 80°, mp below −130°, a 50/50 mixt of the cis and trans isomers. Prepd by fluorinating biguanide sulfate with nitrogen diluted fluorine and a sodium-magnesium fluoride mixt at 0°. Proposed as an oxidizer for proplnts. Should be treated with extreme caution. May readily detonate into CF_4 and N_2. Do not let vapors contact mercury

Refs: 1) Beil, not found 2) J.J. Hockstra, USP 3361815(1968) & CA **68**, 41777(1968)

Octafluoroaminomethylguanidine, $F_2N.C(:NF).NF.CF_2.NF_2$; mw 232.04, N 24.14%, FB 0.0% (See preceding entry); colorless liq, bp 55°, mp below −130°, vap pres 124mm at 10°. Prepd by fluorinating cyanoguanidine with nitrogen diluted fluorine and a sodium-magnesium fluoride mixt at 0°. Proposed as an oxidizer for proplnts. Extremely expl and especially sensitive when undergoing solid-liquid phase transition. Best handled with a CF_2Cl_2 slush bath at −130 to −145°. Do not let vapors contact mercury

Refs: 1) Beil, not found 2) D.A. Rausch & J.J. Hoekstra, JOC **33**(6), 2522–3(1968) & CA **69**, 26687(1968) 3) J.J. Hoekstra, USP 3367968(1968) & CA **69**, 86417(1968)

Pentafluoroguanidine, $F_2N.C(:NF).NF_2$; mw 149.02, N 28.19%, FB +12.8% (See next to last entry); gas, bp −1 to +2° (by extrapolation of vapor pressure data), sp gr 1.51 at 0°, mp −148 to −146°, dec rapidly at 243°. Prepd by fluorination (nitrogen diluted) of guanidine in the presence of sodium-magnesium fluoride mixt at 0°. Very shock and spark sensitive, extremely expl below the bp, may explode during liq-solid or solid-liq transition. Reacts with mercury, hydrolyzes rapidly at RT, but is stable in dry air. Best handled, in liq state, by use of a CF_2Cl_2 slush bath just above the mp. Detonates forming CF_4, N_2, F_2 with a *heat of expln* of −1659cal/g; *heat of formation* is +23.0kcal/m.

Proposed as an oxidizer for proplnts
Refs: 1) Beil, not found 2) R.A. Davis & K.O. Groves, USP 3228936(1966) & CA **64**, 9603(1966) 3) R.A. Davis et al, JOC **32**(5), 1662–3(1967) & CA **67**, 11161(1967) 4) G.C. Sinke et al, JChemPhys **47**(5), 1852–54(1967) & CA **67**, 103347(1967) 5) R.L. Rebertus et al, AnalChem **39**(14), 1867–78(1967) & CA **68**, 18470(1968) (Separation of gaseous organic fluoro nitrogen-contg compds by liq column chromatography) 6) R.J. Koshar et al, JOC **32**(12), 3859–64(1967) & CA **68**, 21461(1968) 7) R.L. Rebertus & B.W. Nippoldt, JOC **32**(12), 4044–45 (1967) & CA **68**, 21462(1968) (Organic fluoronitrogens. VIII. Hydrolytic reactions of tetrafluoroformamidine and pentafluoroguanidine) 8) J.L. Kroon & R.A. Davis, USP 3354011(1967) & CA **68**, 31681(1968) 9) R.L. Rebertus & P.E. Toren, JOC **32**(12), 4045–48(1967) & CA **68**, 39052(1968) (Organic fluoronitrogens. IX. Oxidation-reduction reactions of tris (difluoramino)fluoromethane, tetrafluoroformamidine, and pentafluoroguanidine) 10) C. E. Merrill, USP 3372192(1968) & CA **69**, 2530(1968) & CA **70**, 11160(1969) 11) D.A. Rausch & J.J. Hoekstra, JOC **33**(6), 2522–23(1968) & CA **69**, 26687(1968) 12) D.L. Esmay & G.W. Kotteng, USP 3450762(1969) & CA **71**, 49355(1969) (Prepn from ammeline)
Note: It is advisable for anyone contemplating prepn or use of pentafluoroguanidine to examine all of these refs as a minimum

Tetrachloroiodoguanidine, $H_2N.C(:NH).NH_2.Cl_4I$; mw 327.79, N 12.80%, OB (to Cl_2 and $\frac{1}{2}I_2$) −22.0%, orn-yel prisms, mp 163°, sol in w (with sl dec), stable in dry air. Prepd by treating an HCl soln of Gu.HCl with chlorine and iodine
Refs: 1) Beil **3**, [71] 2) F.D. Chattaway & F.L. Garton, JCS **125**, 183–8(1924) & CA **18**, 986(1924)

α-Tetrazol-5-yl-guanidine or *5-Guanylaminotetrazole,* $C[.NH.C(:NH).NH_2]:N.N:N.NH$; mw 127.11, N 77.15%; wh crysts, mp over 300°. Prepd by heating cyanamide with 5-aminotetrazole. Its expl props were not investigated
Refs: 1) Beil **26**, [244] 2) R. Stollé et al, Ber **62**, 1126(1929)

α-Tetrazol-5-yl-guanidine Nitrate, $C[.NH.C(:NH).NH_2]:N.N:N.NH.HNO_3$; mw 190.12, N 58.96%, OB −33.7%; wh crysts, explodes mildly on heating over 183°; sol in w; sl sol in alc. Prepd by adding nitric acid to an aq soln of cyanoguanidine and hydrazoic acid
Refs: 1) Beil **26**, [244] 2) R. Stollé & K. Heinz, JPrCh **148**, 218, 220(1937) 3) E. Lieber & G.B.L. Smith, ChemRevs **25**, 260(1939)

2,2,2-Trifluoroethylguanidine Nitrate, $F_3C.CH_2.NH.C(:NH).NH_2.HNO_3$; mw 204.11, N 27.45%, OB −39.2% (to CO_2, CF_2O, HF, H_2O, N_2); crysts, mp 134–6°. Prepd from the *picrate* (below) by treatment with nitric acid. Its expl props were not investigated
Refs: 1) Beil, not found 2) V. Milani et al, JACS **77**, 2903(1955) & CA **50**, 3224(1956)

2,2,2-Trifluoroethylguanidine Picrate, $F_3C.CH_2.NH.C(:NH).NH_2.PA$; mw 370.20, N 22.70%, OB − 60.5% (to CO_2, CF_2O, HF, H_2O, N_2); yel ndls, mp 183° (dec). Prepd from *1-nitro-3-(2,2,2-trifluoroethyl)guanidine* by hydrogenolysis followed by treatment with PA. Its expl props were not investigated
Refs: 1) Beil, not found 2) V. Milani et al, JACS **77**, 2903(1955) & CA **50**, 3224(1956)

Guanidinium 3,5-bis or di(nitramino)-as-triazole. See under Diaminotriazoles, Vol **5**, p D1149-R

Guanidinium Dinitrocyanomethylide, $[(H_2N)_3C]^+(O_2N)_2CCN^-$; mw 190.12, N 44.21%, OB −42.1%; crysts, mp 166–7°. Prepd by treating guanidine carbonate in aq alc with ethyl dinitrocyanoacetate at RT. Proposed as proplnt ingredient
Refs: 1) Beil, not found 2) G.O. Parker, USP 3415867(1968) & CA **70**, 57206(1969)

Guanidinium 1,1-dinitroethylide, $[(H_2N)_3C]^+(O_2N)_2CCN_3^-$; mw 179.14, N 39.11%, OB −58.1%; crysts, mp 97°(dec); sol in 5%, wt/wt, w at 20°. Prepd by treating guanidine carbonate with K dinitroethylide in methanol
Refs: 1) Beil, not found 2) L. Zeldin & H. Shechter, JACS **79**, 4708–16(1957) & CA **52**, 2735(1958)

Guanidium 5-dinitromethyltetrazolide, $[(H_2N)_3C]^+(O_2N)_2C.C{:}N.N{:}N.NH^-$; mw 233.15, N 54.08%, OB −37.7%; yel crysts, mp 164–4.5° (dec). Prepd by treating guanidine carbonate with 5-dinitromethyltetrazole. Its expl props were not investigated

Refs: 1) Beil, not found 2) F.J. Einberg, JOC **29**(7), 2021–24(1964) & CA **61**, 9490(1964)

Guanidinium 3,5-dinitro-as-4H-triazolide; $[(H_2N)_3C]^+N.C(NO):N.N:C(NO_2)^-$; mw 218.13, N 51.40%, OB −36.7%; lt yel crysts, mp 225–8° (dec). Prepd by treating guanidine carbonate with 3,5-dinitro-as-4H-triazole in acet. The salt exhibits an impact sensitivity (2kg weight) of over 320cm, and is suggested for use as a press-loaded expl

Refs: 1) Beil, not found 2) H.P. Burchfield & D.K. Gullstrom, USP 3054800(1962) & CA **58**, 10221(1963)

Guanidinium Fulminuride, $[(H_2N)_3C]^+NCC(NO_2)C(O)NH_2^-$; mw 188.15, N 44.68%, OB −76.6%; crysts, mp 221–3°(dec). Prepd by treating guanidine hydrochloride with fulminuric acid. Its expl props were not investigated

Refs: 1) Beil, not found 2) J.A. Carbon, JOC **26**, 455–61(1961) & CA **55**, 13434(1961)

Guanidinium Hydrazine Decaborohydride, $[(H_2N)_3C]^+[N_2H_4.B_{10}H_{13}]^-$; mw 213.34, N 32.83%; crysts. Prepd by adding decaborane, $B_{10}H_{14}$, to an aq soln of guanidine and hydrazine, no props given. Compares unfavorably with the triaminoguanidine analog as a fuel in rocket proplnts

Refs: 1) Beil, not found 2) E.A. Takacs, USP 3281219(1966) & CA **66**, 4569(1967) (Hydrazinates and method of preparing them)

Guanidinium Hydrazinium Dihydrazoate, $[(H_2N)_3C]^+H_2NNH_3^-(N_3)_2^=$; mw 177.17, N 87.06%, OB −67.8%; crysts. Prepd by heating a mixture of guanidine & hydrazine sulfates with sodium azide in methanol; no props given

Ref: Beil, not found

Guanidinium Hydrazoate or **Guanidinium Azide,** $[(H_2N)_3C]^+N_3^-$; mw 147.14, N 85.71%, OB −70.7%; colorless prisms, mp 93.5°(46°, Ref 3); 159.2g sol in 100g w at 20°; sol in aq alc & acet. Prepd by reacting with guanidine-HCl, AgN_3; with guanidine.H_2SO_4, $Ba(N_3)_2$; or with guanidine.H_2CO_3, HN_3. Deliquesces slowly when exposed to room air. Its expl props were not investigated

Refs: 1) Beil **3**, [160] 2) J. Craik et al, JACS 56, 2380–1(1934) & CA 29, 700(1935) 3) A. Cirulis & M. Straumanis, JPrCh **161**, 65–76(1942) & CA **37**, 5023(1943)

Guanidinium 5-nitraminotetrazolide. See Vol 1, p A260-L

Guanidinium 5-(3-nitro-1-guanidino) tetrazolide, $[(H_2N)_3C]^+O_2N.NH.C(:NH).NH.C{:}N.N{:}N.N^-$; mw 231.18, N 66.67%, OB −58.9%; wh rosettes, dec 240–2° in a hot bath. Prepd by mixing in aq alc: guanidine carbonate, 5-aminotetrazole, and 1-methyl-3-nitro-1-nitrosoguanidine. Suggested as a substitute for NGu in proplnts

Refs: 1) Beil, not found 2) J. Cohen et al, USP 3073731(1963) & CA **58**, 11164(1963)

Guanidinium 3,5,7-trinitrotropolonate, $[(H_2N)_3C]^+C(:O).C(.O^-):C(NO_2).CH:C(NO_2)$; mw 361.23, N 34.90%, OB 59.8%; crysts, mp 253°(dec). Prepd by heating guanidine sulfate in methanolic $NaOCH_3$ with a 1/1 complex of tropone and 3,5,7-trinitrotropolone. Its expl props were not investigated

Refs: 1) Beil, not found 2) T. Nozoe et al, BullChemSocJapan **36**, 168–73(1963) & CA **59**, 6308(1963)

$N^{2,6}$-Diguanidinomelamine Dihydrochloride, $C(.NH.Gu):N.C(.NH.Gu):N.C(.NH_2):N$. 2HCl; mw 313.15, N 53.67%; crysts, mp 320–2°. Prepd by reacting cyanoguanidine with phenol in HCl. Treatment with HNO_3 gives the **dinitrate,** mw 366.26, N 53.54%, OB − 48.1%; mp over 360°; treatment with PA gives the **dipcrate,** mw 698.445, N 36.10%, OB −66.5%; mp 276–7°. Their expl props were not investigated

Refs: 1) Beil, not found 2) S. Bando et al, YukiGoseiKagakuKyokaiShi **28**(5), 521–5(1970 & CA **73**, 45477(1970)

1,4-Dihydro-2-amino-4,4-dimethyl-6-guanidino-s-triazine Dinitrate,
$C(.NH_2):N.C(CH_3)_2.N:C(.Gu).NH \cdot 2HNO_3$;
mw 309.24, N 40.78%, OB –69.9%, crysts, mp 228–9°. Prepd by treatment of the *monopicrate*, mp 231°, with nitric acid. The **picrate** had been prepd by heating with AN at 180° 1,4-dihydro-2-amino-6-cyanamido-4,4-dimethyl-s-triazine, then adding ammonium picrate. Their expl props were not investigated
Refs: 1) Beil, not found 2) K. Odo & E. Ichikawa, JapP 70' 15265 (1970) & CA **73**, 45547 (1970)

1,4-Dihydro-2,6-bis(guanidino)-1-guanyl-4-guanylimino-s-triazine. Trihydrochloride,
$C(.Gu):N.C(:Gu).N:C(.Gu).N.C(:NH).NH_2 \cdot 3HCl$;
mw 361.63, N 46.49%; crysts, mp over 360°. Prepd by heating at 70° a mixt of dicyandiamide, cyanamide dihydrochloride, and phenol. Treatment with nitric or picric acids gave the corresponding **nitrate** or **picrate** (number of acid moieties not indicated), mp's over 360°. Their expl props were not investigated
Refs: 1) Beil, not found 2) K. Odo & E. Ichikawa, JapP 71'00031 (1971) & CA **74**, 141885 (1971)

Guanidinoformylazide Hydrochloride (Guanidin Carbonsaure Azid in Ger),
$H_2N.C(:NH).NH.C(:O).N_3 \cdot HCl$; mw 128.09, N 51.06%, OB –53.5% (to $\frac{1}{2}Cl_2$); crysts, mp 157°; sol in w; insol in organics; explodes on heating. Prepd by treating 1-amino-3-guanylurea dihydrochloride with N_2O_5 in chlf. Its expl props were not investigated
Refs: 1) Beil **3**, 130 2) J. Thiele & E. Uhlfelder, Ann **303**, 112 (1898)

N^2-Guanidinomelamine Dihydrochloride,
$C(.NH.Gu):N.C(.NH_2):N.C(.NH_2):N \cdot 2HCl$;
mw 256.10, N 49.22%; crysts, mp 298°. Prepd by reacting cyanoguanidine with phenol in HCl. Treatment with nitric acid gives the **dinitrate**, mw 309.20, N 49.84%, OB – 38.8%, mp 257°; treatment with PA gives the **monopicrate**, mw 412.29, N 40.77%, OB –73.8%, mp 290.5°. Their expl props were not investigated
Refs: 1) Beil, not found 2) S. Bando et al, YukiGoseiKagakuKyokaiShi **28**(5), 521–5 (1970) & CA **73**, 45477 (1970)

2,4,6-Triguanidino-s-triazine or **$N^{2,4,6}$-Trigyanylmelamine Trihydrate,**
$C(.Gu):N.C(.Gu):N.C(.Gu):N \cdot 3H_2O$; mw 306.29, N 54.88%. Prepd by treating with w (at pH over 10) 1,4-dihydro-2,6-bis(guanidino)-1-guanyl-4-guanylimino-s-triazine trihydrochloride (See above); no props given and expl props were not investigated
Refs: 1) Beil, not found 2) K. Odo & E. Ichikawa, JapP 71' 00031 (1971) & CA **74**, 141885 (1971)

Guanite (Fr). See Nitroguanidine under Guanidine

N,N'-Bis(guanyl)-N,N'-diaminoethylenediamine Dihydrazoate,
$[CH_2.N(.NH_2).C(:NH).NH_2]_2 \cdot 2HN_3$; mw 260.27, N 75.38%; crysts, mp 209–13° (dec). Prepd from the *dibromide*, mp 299–301°, by treatment with a basic ion exchange resin followed by treatment with HN_3. The dibromide was prepd from N,N'-diaminoethylenediamine and s-methyl isothiourea hydrobromide in methanol at RT. The corresponding bis(diaminoguanyl) compd was found more useful as a proplnt fuel
Refs: 1) Beil, not found 2) E.T. Niles et al, USP 3355491 (1967) & CA **68**, 21557 (1968)

N,N'-Bis(guanyl)-trimethylenediamine or *Methylene bis(guanidinomethane)*,
$CH_2(.CH_2.NH.C(:NH).NH_2)_2$; mw 158.21, N 53.16%; hygr crysts, mp 135°; sol in alc & w. Prepd by treating methylene bis(aminomethane) with s-methyl isothiourea sulfate. More recently isolated as the **dipicrate** salt by heating a mixt of cyanamide, methylene bis(aminomethane), and methylene bis (aminomethane p-toluenesulfonate), then treating with PA; for the dipicrate: mw 616.42, N 27.27%, OB –77.9%, mp 242°. Their expl props were not investigated
Refs: 1) Beil **4**, [700] 2) M. Schenck & Kirchhof, ZPhysiolChem **158**, 107 (1926) 3) B. Adcock et al, JCS **1961**, 5123 & CA **56**, 11577 (1962)

Guanyl Azide Derivatives and Salts. See Azidoformamidine under Formamidine in Vol **6**, pp F168-L to F169-R

Guanyldiazoguanyl Tetrazene. See Guanylnitrosaminoguanyl Tetrazene below

Guanylglycine. Same as Glycocyamine

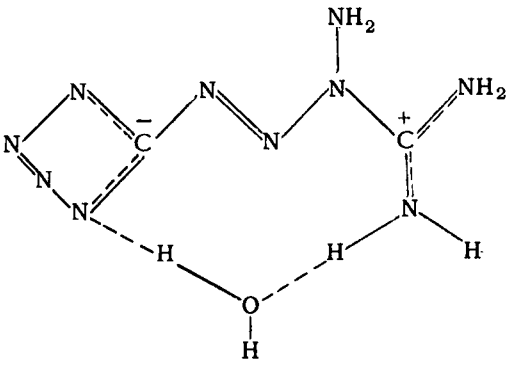
Guanylguanidine. See Biguanide in Vol **2**, p B114-L

Guanylhydrazine. See Aminoguanidine in Vol **1**, p A210-L

Guanylnitrosaminoguanyl Tetrazene or **TETRACENE,** also known as *4-Guanyl-1-(a-tetrazol-5yl)-tetrazene* or *Guanyldiazoguanyl Tetrazene.* (It must not be confused with naphthacene which is also called Tetracene); (Called Buzylene, rather than Tetrazene, in Ref 2, but same structure is meant; in recent literature the term "guanyl" has been replaced by "amidino", and then "carboxamidine"). Patinkin et al (Ref 11) claimed that the Tetrazole Hydrate, rather than the Nitrosaminoguanyl, was the actual structure, but both structures continue in use. Patinkin also prepd a **nitrate** salt, mp 93–94° (dec) of the (claimed) Tetrazole structure but with the 1 and 4 positions of the Tetrazene interchanged. Hofmann et al, the original preparers (Ref 2), also seemed to believe they had the Tetrazole and Nitrosaminoguanyl structure as separate compds. Its formula given in Davis (Ref 10) and AMCP–706–177 is

Its hydrate contns $3H_2O$; mw of anhyd compd 188.15, N 74.47%, OB to CO_2 −57.6%, OB to CO ca −43%

A recent study of the structural analysis by single cryst methods (Refs 14 & 15) shows that Tetracene is the zwitterion (guanidinium) of 1-Amino-1-[(1H-tetrazole-5-yl)azo]- guanidine Hydrate of structure:



in contrast to the structure previously assigned by Patinkin et al (Ref 11)

Tetracene exists as colorless or pale yel ndls; mp decomp ca 142° and explodes at 160° in 5 secs (vs 190° for MF); explodes in a flame w/o much noise but producing much bk smoke; apparent d only 0.45, but yields a pellet of d 1.05 when compressed at 3000 psi. It is practically insol in w, alc, eth, acet, benz, CCl_4 and ethylene dichloride; sol in dilute nitric acid; decompd by boiling w with formation of $2N_2$ per mole; can easily be dead-pressed (Ref 10)

Tetracene was first prepd in 1910 by Hofmann et al (Ref 2) and then studied by them (Ref 3), but the most extensive studies were done by Rinkenbach & Burton (Ref 4) and results of their experiments are given by Davis (Ref 10) and in AMCP 706–177 (Ref 12)

Davis (Ref 10, p 449) describes in detail a method of prepn starting with 35g of aminoguanidine bicarbonate, 2500ml distd w, 15.7g of glac AcOH and 27.6g of Na nitrite.

One lab method described in AMCP - 706–177 is not in the literature: Tetracene was prepd by dissolving 5g of aminoguanidine dinitrate in 30mm of distd w, cooling to 0°C and mixing with a soln 2.5g of Na nitrite in 15ml of w. While maintaining the temp at 10°, 0.5g of glac AcOH was added causing pptn of Tetracene. After allowing the slurry to stand overnight (or longer), the ppt was washed by decantation with several portions of w and then transferred to filter to be washed more thoroughly with w using suction. Then the product was dried in air at RT and transferred for storage in a bottle provided either with a cork or rubber stopper

Another method of prepn listed in AMCP 706–177 is from aminoguanidine sulfate and Na nitrite in distd w

Following are expl props of Tetracene as given in Refs:

Brisance, by Sand Test. 0.4g initiated by No 6 cap crushed 28.0g sand, vs 23.4g by MF or 48.0g by TNT

Detonation Rate. not found

Explosion Temperature. 160° in 5 secs

Gas Volume. 1190cc/g

Heat of Explosion. 658cal/g

Heat Test at 100°. loss 23.2% in 1st 48 hrs, 3.4% in 2nd 48 hrs; no expln in 100 hrs

Hygroscopicity. only 0.77% gain at 30° & RH 90%

Impact Sensitivity. BM App, 2kg wt 7cm, vs 5cm for MF

Impact Sensitivity. PicArsnApp, 8 oz wt 8 inches, vs 9–10 inches for MF (Davis, p 449)

Initiating Efficiency. not efficient when used alone for initiating HE's, but is as efficient as MF when initiated externally (Davis, p 450)

International Test at 75°. % loss in 48 hrs 0.5, vs 0.18 for MF

Loading. pressed

Power by Trauzl Test. 61% of TNT, vs 51% by MF

Sensitivity to Electrostatic Discharge, in joules. 0.010 for unconfined and 0.012 for confined

Sensitivity to Initiation. min detong chge 0.40g MF

Storage. wet

Uses: Can be used in detonators when initiated by another primary expl and functioning as an intermediate booster or when mixed with another primary expl to increase the sensitivity of the latter to flame or heat. Its mixture with LA was patented by Dynamit AG (Ref 8) for use in explosive rivets. Tetracene can also be used in primer caps where as little as 2% in the compn results in improved uniformity of percussion sensitivity

The following Ger WWII priming mixts contg Tetracene are listed in Ref 11a, p Ger 197-L:

I. Priming Mixture No 30/40, used for rifle and pistol cartridges: Tetracene 3, Pb styphnate 40, Ba nitrate 42, Ca silicide 10 & 0b dioxide 5%

II. Duplex-Cap Mixture for use in 20mm & 37mm, as well as in some larger shells, consisted of 0.30g of Pb azide 92.5 and Tetracene 7.5% pressed at 100kg/sq cm over 0.05g of unwaxed PETN pressed at 500kg/sq cm

III. Priming Mixtures used for pistol and rifle cartridges: Tetracene 2–3, Pb azide 30–35, Ba nitrate 40–45, Ca silicide 6–12, Pb peroxide 5–8 and Sb sulfide 6–9%

IV. *Sinoxydsätze*, listed on p Ger 179-R, were developed before WWII as replacement of MF mixts. They were claimed to be non-corrosive and did not erode barrels of firearms. Their compn was: Pb styphnate 25–55, Tetracene 1.2–5.0, Ba nitrate 25–45, Pb dioxide 5–10, Sb sulfide 0–10, Ca silicide 3–15 and powdered glass 0–5%

Refs: 1) Beil **3**, (60), [103] & {240} 2) K.A. Hofmann et al, Ber **43**, 682–83 & 1087–91 (1910) 3) K.A. Hofmann et al, Ber **44**, 2496 (1911) & Ann **131** (1911) 4) W.H. Rinkenbach & O. Burton, ArmyOrdn **12**, 120 (19319 5) A. Stettbacher, Nitrocellulose **8**, 141 (1936) 6) F. Grottanelli, Chim e Industria **18**, 232 (1936) 7) G.B.L. Smith, ChemRev **25**, 214 (1939) (Chemistry of aminoguanidine and related compds) 8) Dynamit AG, vorm Alfred Nobel & Co, BritP 528299 (1940) (Expl rivets using Tetracene) 9) Clift & Fedoroff, Vol **1** (1943), p G6 (Guanylnitrosaminoguanyltetrazene) 10) Davis (1943), 446–50 (Tetracene) 11) S.H. Patinkin et al, JACS **77**, 562–67 (1955) & CA **50**, 972 (1956) 11a) B.T. Fedoroff et al, PATR **2510** (1958), pp Ger 196-R & Ger 197-L:

Tetracene (Tetrazen). The following Ger method of prepn used during WWII is described. Utilizing the reactor provided with air agitation, to a soln contg 4.0kg of Na nitrite in 60 liters of water and 1.5 liters of normal AcOH preheated to 50°, was added gradually and with air agitation 40 liters of an aq soln of 5.3kg of aminoguanidine sulfate. The addn took 1 hr. After stirring the slurry for an addnl hr at 50° and for an hr at 20°, the reactor was tipped and the contents caught on the filter cloth made

of horse hair. After thorough washing of ppt with water it was placed in plastic buckets and transferred to the storage area. For Tetracene, which had to be dried prior to use, the water washing on the filter cloth was followed by rinsing with 96% ethanol contg some methanol. The yield of dry product was 2.6–2.7kg per 4.0kg of aminoguanidine sulfate 12) Col,"Engineering Design Handbook. Properties of Explosives of Military Interest", US Army Materiel Command Pamphlet, **AMCP 706-177** (1967), 324–26 and (1971), 324–26 13) Sax (1968), p 1154(Tetrazene) 14) J.R.C. Duke, Chem-Commun **1971**, 2–3 15) I. Dunstan, Chemistry in Britain **7**, 66(1971) 16). Cond-ChemDict (1971), p 861-R (Tetrazene)

Guanylnitrosaminoguanyltetrazene, TETRACENE or [*1-(5-Tetrazolyl)-4-guanyltetrazene hydrate*]. US Military Specification MIL-T-46938A (MU) (Ref 3) requirements and tests for material intended for use in military primer compositions

Requirements:

3.3 *Color and Appearance.* Tetracene shall be white to light yellow and under microscope it shall appear as needle-shaped crysts

3.4 *Melting and Explosion Point:* 130±2°

3.5 *Granulation:* 100% shall pass thru US No 45 sieve

3.6 *Specific Gravity:* 1.65±0.05

3.7 *Heavy Metals:* must be absent

3.8 *Nitrogen Content:* 74.5±1.0%

Test Methods and Procedures

4.4.1 *Color and Appearance.* Transfer a portion of about 10g of the wet sample as received to a Büchner funnel and apply suction until the sample is almost dry. Spread the sample on a watch glass and place in a desiccator over concd sulfuric acid for 24 hours or in an oven at 70–75°C for at least 90 mins. Use this sample for all detns where a dry sample is required. Take four portions of about 0.5mg each from different places in the sample and spread them over an area of about 2cm square on a glass microscope slide. Observe the color of the material and then examine it under a microscope with reflected light using a magnification of 350 times

4.4.2 *Melting Point and Explosion Point.* Weigh a capillary tube (1.5 to 2mm in diam) and add a dry sample of Tetracene until about 0.5 to 1.0mg is present. Put on a safety mask and detn the mp in a Vanderkamp apparatus (Scientific Glass Apparatus Co Catalog 59, M-1945, Bloomfield, New Jersey). Heat the capillary tube in the inclosed airspace. Regulate the electrical heating system so that the rise in temp is one degree C every 3 mins. Observe the capillary tube thru the magnifying glass, which is part of the apparatus, and note when the substance melts and when the substance explodes

4.4.3 *Granulation.* Transfer about 30g of the wet sample, as received, to a Büchner funnel about seven cm in diam and apply suction for two mins. Weigh a 10g portion of this damp sample and transfer it to a tared 3-inch No 45 US Standard sieve, which has been placed in a porcelain evaporating dish containing sufficient water to cover the wire portion of the sieve. Shake the sieve for a few minutes in such a manner that the Tetracene is below the surface of the water and any material passing thru the sieve is caught in the evaporating dish. When most of the Tetracene has passed thru the sieve, run a stream of water thru the sieve while brushing the residue lightly with a soft, camel-hair brush. When no more material appears to pass thru the sieve, rinse that remaining on the sieve with 95% ethyl alcohol or reagent grade methanol, isopropanol, or mixt thereof and dry the sieve and contents on a steam bath. Place the sieve in an oven at 70°–75°C for 30 mins, cool in a desiccator and weigh. Calculate the gain in weight as percent material retained on the No 45 sieve

4.4.4 *Specific Gravity.* Weigh a dry 10ml pycnometer. Fill it with water at 30°C and weigh again. Empty and dry pycnometer, add 0.2–0.4g of dry Tetracene and weigh again. Add about 5ml of kerosene and put under vacuum in a vacuum desiccator for a few minutes to remove air bubbles. Fill pycnometer with kerosene at 30°C and weigh. Calculate as follows:

$$\text{Sp gr of Tetracene} = \frac{W}{A - \frac{(B-C)}{S}}$$

where: W = wt of sample
A = wt of water contd in bottle when filled
B = wt of bottle + Tetracene + kerosene
C = wt of bottle + Tetracene
S = sp gr of kerosene

4.4.5 *Heavy Metals.* To test for the presence of heavy metals, treat a 0.3g dry sample of Tetracene with 10ml of concd nitric acid and 5ml of concd sulfuric acid, evaporate to strong fumes until the soln is water white. Cool, dilute to 150ml with w and treat with hydrogen sulfide. No detectable ppt will be present

4.4.6 *Nitrogen Content.* A dry sample of 2mg of Tetracene will be used for the nitrogen detn by the Micro-Dumas Method. Detailed instruction for performing this analysis can be found in standard tests on organic analysis, for example, "Quantitative Organic Microanalysis" by A. Steyermark, obtainable from McGraw-Hill Book Co, Inc, 330 W 42nd St, New York, NY

Tetracene can be identified by wetting a 0.25g sample with 5ml of 10% NaOH soln, and warming the mixt on a steam bath until it is dissolved. Note if odor of ammonia is present. Cool the soln, and add 1 ml of 5% Cu acetate soln. The appearance of a bright blue ppt indicates Tetracene (Ref 1, p 1346)

For a primer compn contg Ba nitrate, normal or basic Pb Styphnate, Tetracene, Sb sulfide & Pb Azide, Tetracene content is detd by washing the residue from the styphnate detn several times with alc, aspirating, drying at 55–65° for 30 mins, cooling and reweighing. Using a jet of water, transfer the dry residue to a 125-ml beaker, add 25ml water, and boil for 5 mins. Filter thru the crucible previously used, wash the residue with boiling water and then alc. Aspirate, dry & reweigh as before. The loss in wt by water extraction represents Tetracene (Ref 1, p 1370)

Refs: 1) StdMethodsChemAnalysis **2B** (1963), pp 1346 & 1370 2) Frank Pristera, "Explosives" in Vol 12 of "Encyclopedia of Industrial Chemical Analysis", J. Wiley, NY (1971), 415–16 (Tetracene) 3) US Specification MIL-T-46938A (April 1971) (Tetracene)

Guanylnitrosaminoguanyltetrazene or **Tetracene Salts.** It forms numerous salts, most of which are explosive. Several of them are listed in Beil and other refs

For example, Hofmann et al (Ref 2) prepd an HI_3 salt, black cubic crysts, explode mildly on heating, violently on contact with silver nitrate and nitric acid; also a **Silver** salt, yel crysts, very expl; the same salt is described in Davis (Ref 3, p 447)

Davis lists a **Copper** salt, a **Nitrate, Perchlorate** and **Picrate** (Ref 3, pp 469–70). He considers the perchlorate especially interesting. It dissolves in strong HCl and ether ppts the hydrochloride from the soln. Both the Perchlorate and the Picrate explode violently from heat and from shock

Refs: 1) Beil **3**, (60–1), [103] & {240} 2) K.A. Hofmann et al, Ber **43**, 1091 (1910) & CA **4**, 2465 (1910) 3) Davis (1943), 447–48

Guanylpentafluorodiaminomethane, $H_2N.C(:NH).CF.(NF_2)_2$; mw 178.065, N 31.46%, OB −27.0% (to CO_2, F_2CO, 3HF, $2N_2$); yel visc oil, vap press 1 mm at 25°. Prepd from a mixt of pentafluoro bisaminoacrylonitrile, ammonia, fluorotrichloromethane, and methyl ether at −110 to +25°. Probably a strong oxidizing agent; may be shock sensitive

Refs: 1) Beil, not found 2) R.J. Koshar, USP 3654341 (1972) & CA **76**, 139957 (1972)

Guanyltetrazolyl Tetrazene. See Guanylnitrosaminoguanyl Tetrazene or TETRACENE

Guanylurea, Dicyandiamidine or Carbamylguanidine and Derivatives. See Vol **5**, p D1217-R

Trifluoroguanylfluoride or **Tetrafluoroformamidine,** $F_2N.C(:NF).F$; mw 116.02, N 24.14%, FB 0.0% (See under Pentafluoroguanidine); gas, bp ca −30°, dec at 208°. Prepd by fluorination of guanylurea sulfate (nitrogen dilution)

at −10°. The compd is assumed to be the sym isomer, shows ready hydrolysis in w, is shock sensitive, and should be considered a powerful expl and oxidizing agent
Refs: 1) Beil, not found 2) R.J. Koshar et al, JOC **32**(12), 3859–64(1967) & CA **68**, 21461(1968) 3) R.L. Rebertus & B.W. Nippoldt, JOC **32**(12), 4044–45(1967) & CA **68**, 21462(1968) (Organic fluoronitrogens. VIII. Hydrolytic reactions of Tetrafluoroformamidine and Pentafluoroguanidine) 4) R.L. Rebertus & P.E. Toren, JOC **32**(12), 4045–48(1967) & CA **68**, 39052(1968) (Organic fluoronitrogens. IX. Oxidation-reduction reactions of tris (difluoramino) fluoromethane, Tetrafluoroformamidine and Pentafluoroguanidine) 5) R.J. Koshar & D.R. Husted, USP 3461162(1969) & CA **71**, 101290(1969)

Guar Gum or **Guar Flour.** Light-grey pdr dispersible in hot or cold w; has 5–8 times the thickening power of starch. It is the ground endosperm of *Cyanopsos tetragonoloba*, cultivated in Pakistan as livestock feed. The water-sol portion of the flour (85%) is called guaran and consists of 35 parts galactose, 63 mannose and 5–7 protein. Used as a binding agent in expl compns; also in paper manuf, atomic metal processing, etc

As examples of expls contg guar gum one may cite Blasting Explosive listed as Ref 117 on p D1658-R of Encycl, Vol **5**. The gum is used with borax as its cross-linking agent. Another example is explosive slurry contg 1% guar gum listed in Ref 171, p D1662-R. The slurried Blasting Expl contg 1.9% guar gum is listed in Ref 178 on p D1663. Expl compn listed as Ref 214 on p D1666-R contd 1% guar gum. Brit safety expl listed as Ref 223 on p D1667-R contd 1% guar gum. Expl compn contg 1% guar gum is listed as Ref 277 on p D1674-L and many other compns some of them contg less than 1% guar gum (Other gums are described under GUM)

Gudolpulver. A German flashless (cool) propellant. See Vol **5**, pp D1537-R to D1538-R and PATR **2510**(1958), pp Ger 81-L & R. Also in Vol **6**, p E116-L, under EROSION OF GUN BARRELS

Gudron. Russ for Tar or Pitch

Guerilla Incendiary Mixture. According to Stettbacher (Ref) the following expl incendiary mixt was used by guerillas during WWII. It was prepd by dissolving about 30.5 parts of yellow phosphorus in some carbon disulfide and adding to it about 62.5 parts of powdered $KClO_3$. When this liquid was poured on some object (such as tank), self-ignition and even explosion took place just as soon as the carbon disulfide evaporated
Ref: A. Stettbacher, Protar **40**, 160(1944)

Guerrilla Warfare. This term refers to military operations conducted by informal forces which operate within territory nominally controlled by the enemy and which have no permanent bases or other permanently defended territory. Guerrilla warfare is usually thought of as small-scale and independent operations, as in insurgency or revolutionary war situations. Geurilla warfare can also be a secondary adjunct to ordinary military operations, as in the case of the Palestinian commandos

Guerrilla warfare is based on temporary concentrations on the offensive, followed by dispersion on the defensive. This may involve going clandestine (using concealment by natural cover, eg forests) or covert (disguise by assuming a civilian cover). The actual tactics are conventional rule-book ones for an inferior force: avoidance of battle, ambuscades, and the slow erosion of superior forces by repeated attacks on outposts & supply lines. The key factor in guerrilla warfare is the source of supplies, since the force lacks either a home territory or supply lines to it. Where subversion does not work and where the home base does not exist or cannot overcome the logistic problem, guerrillas cannot survive as effective fighting forces because they have to acquire food supplies by using force against the local population, which is politically inefficient & tactically inefficient

Ref: E. Luttwak, "A Dictionary of Modern War", Harper & Row, NY (1971), pp 102–03
NOTE: See also the following books on Guerrilla Warfare:
1) Anon, "Guerrilla Warfare and Special Forces Operations", US Dept of the Army, Washington, DC (1961)
2) Che Guevara, "On Guerrilla Warfare", F.A.Praeger, NY (1961)
3) Brig Gen S.B. Griffith, "On Guerrilla Warfare", F.A. Praeger, NY (1961)
4) Col V. Ney, "Notes on Guerrilla War. Principles and Practices", Command Publications, Washington, DC (1961)
5) F.M. Osanka, "Modern Guerrilla Warfare", The Free Press of Glencoe, NY (1962)
6) Lt Col T.N. Greene, "The Guerrilla – and How to Fight Him", F.A. Praeger, NY (1962)
7) O. Heilbrunn, "Partisan Warfare", G. Allen & Unwin Ltd, London (1962)
8) P. Paret & J.W. Shy, "Guerrillas in the 1960's", F.A. Praeger, NY (1962)
9) Staff, "Studies in Guerrilla Warfare", United States Naval Institute, George Banta Co Inc, Mdnasha, Wis (1962)
10) C.W. Thayer, "Guerrilla", Michael Joseph, London (1963)
11) Lt Col J.J. McCuen, "The Art of Counter-Revolutionary War", Faber & Faber, London (1966)
12) Brig Gen S.B. Griffith, "Mao Tse-Tung Basic Tactics", F.A. Praeger, NY (1966)
13) J. Paget, "Counter-Insurgency Operations: Techniques of Guerrilla Warfare", Walker & Co, NY (1967)

Guhr, Kieselguhr, Diatomite, Diatomaceous Earth (DE) or **Infusorial Earth.** A soft earthy rock composed of siliceous skeletons of small aquatic plants called "diatoms". Light-colored solid resembling chalk or dried clay in appearance; sp gr 1.9–2.35 (true), 0.15–0.45 (apparent); insol in acids except HF; sol in strong alkalies; able to absorb up to 4 times its own wt in water or in NG. Used in filtration, clarifying, and decolorizing of liquids and (in some countries except USA) as an absorbent ("dope") for NG in so-called *Guhrdynamite* (See under DYNAMITES in Vol **5**, p D1595-L)

Refs: 1) CondChemDict (1961), pp 353–54) 2) Anon, "Properties of Materials Used in Pyrotechnic Compositions", **AMCP 706–187**, Washington, DC (1963), pp 117–18 3) Kirk & Othmer **7** (1965), pp 53–63 (Diatomite) 4) CondChemDict (1971), p 274-R (Diatomaceous Earth) 5) US Specification, MIL-D-20550B (Aug 1968) (Diatomaceous Earth)

Guggenheim Process. A process for the manuf of Na nitrate from Chilean nitrate ore, caliche, in which heat is efficiently utilized and handling costs are kept to a minimum
Ref: CondChemDict (1961), p 554-L & (1971), p 429-L

Guhrdynamit or **Dynamit No 1,** also known as *Nobel's Safety Powder.* Explosive consisting of NG 75 absorbed on kieselguhr 25%, invented by Alfred Nobel and patented in England in 1867

Its history is discussed in Vol **5** of Encycl, pp D1588-R & D1589-L and prepn & props on pp D1595-L to D1596-R

Guhrhellhoffit. Ger expl prepd ca 1880 by absorbing with kieselguhr a mixt of Nitrobenzene and fuming nitric acid (See also under Hellhoffit in Vol **7** of Encycl)
Refs: 1) Colver (1918), 143 2) PATR **2510** (1958), Ger 81-R

Guichard's Balance Method for Determination of Stability. The stability of an expl may be judged by the rate at which it loses weight at elevated temps. In the regular procedures, a weighed sample is heated in an oven for a definite length of time, then removed, cooled and reweighed. This gives the loss in weight for a definite time and temp. If it is desired to prep a curve giving the relation between time, loss of wt and temp, heating has to be repeated for another time interval, the sample reweighed, and so on. In order to eliminate the necessity of repeatedly removing the sample from the oven for weighing, Prof M. Guichard at Sorbonne, Paris, France (Refs 1 & 2) constructed a balance with hydrostatic compensation

which permitted the continuous weighing of the sample without removing it from the oven. The temp of the oven can either be kept constant or set to rise at a desired rate per unit of time. This apparatus is described in detail in Ref 3

Instead of a balance with hydrostatic compensation, a chainomatic balance may be used, but the rest of the apparatus should be the same as described by Guichard

Refs: 1) M. Guichard, BullSocChim(France) [4] **39**, 1113(1926) and [5] **3**, 115(1936)
2) Ibid, Ann de Chim **9**, 324(1938)
3) Clift & Fedoroff, "Manual for Explosives Laboratories", Lefax Soc Inc, Philadelphia (1943), Vol **1**, Chap 12, pp 15–19

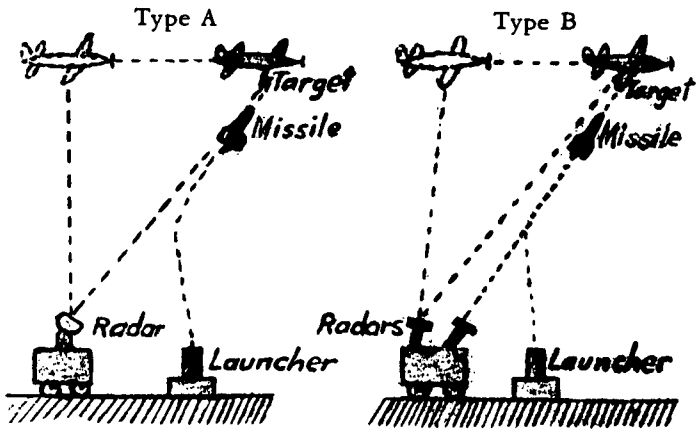
Guidance System of a Missile (See also under GUIDED MISSILES). Accdg to Locke (Ref 4, p 4), it may be defined as a group of components which measures the position of a guided missile with respect to its target and causes changes in the flight path as required. Some of the system elements may be external to the missile located either at the launching site, or enroute to the target or on the target itself

Normally the guidance includes: sensing, computing, directing, stabilizing and servo-control components

Accdg to Gatland (Ref 2, pp 13–16), the post WWII American and European guidance systems may be subdivided into:
A. Beam Rider Control System. With this system a ground radar tracks the target (such as an airplane), while the attacking missile climbs within the cone of a radar beam towards the target. The system is usually considered in conjunction with a self-homing device which monitors the gyro-pilot of missile so that in the final stage of an attack the missile is self-directing. This system is not as good as the B
B. Command Guidance System. With this system one radar tracks the target, while the other tracks the missile. Each radar feeds data into a computer, whereby steering commands are transmitted to the missile

Some of the current Amer guidance systems may be considered as modifications and improvements of German systems developed before and during WWII (See under GUIDED MISSILES, Historical). For this reason we are including below a brief description of German systems, as given in PATR **2510** (Ref 6, pp Ger 83 & Ger 84 and in Refs 1, 3 & 4)

GUIDANCE SYSTEMS



A. Acoustic Homing Devices. These utilized the sound produced by airplane engines as a guiding medium. Two such devices were developed and were intended for guiding the X-4 missile. Both systems received the sounds from two separate entrance ports and determined the direction of the target by comparing the phase of the incident sound front. Phase comparison circuits were used to command the missile to maneuver so that the phase angles became equal. This made the missile point directly at the target. The principal advantage of the acoustic homing missile was the impossibility of jamming its receivers (such as is done with radio controlled guidance systems (Ref 4, pp 602–5)

Accdg to Ref 3, pp 216–19 & 229, the original acoustic homing system was called **Kranich** and the later version **Pudel.** The Pudel fuze was not sufficiently developed to be used in combat, but the Kranich fuze was. The Kranich consisted of a light diaphragm-actuated mechanism which responded to the sound of airplane propellers at a range of 15 meters. It was constructed on the same principle as the Pudel fuze. It was planned to install the Kranich system on some Rheintochter missiles

B. Ballistic Guidance System, also called **Inertial-Gravitation Guidance System.** This was essentially similar to a long-range gunfire guidance. As with a gun for surface fire, a missile such as a V-2 (A-4) was aimed in the desired direction in azimuth and pointed at such a precalculated elevation angle that the projectile would fall to the surface at the correct target range. The V-2 was directed in heading during its burning period by four external and four internal vanes. The external vanes, located in the outer trailing edge of each large fin, created aerodynamic moments, whereas the internal vanes, made of carbon and located to the rear of the motor, varied the direction of thrust of the motor. For control in azimuth, the external and internal vanes were interlocked but they were so connected as to permit separate control in pitch. (Ref 4, pp 36–8 & 583–4)

Accdg to Ref 3, p 211, the V-2 missile was regulated in flight by fins which were positioned by hydraulic servo-mechanisms controlled by an elaborate intelligence system. This system consisted of:

a) Two gyroscopes to provide stability about the three axes of the missile

b) Radio (optional) to provide azimuth control by flying on a beam

c) Radio or integrating accelerometer for tuning the motor at a specific velocity, to provide range control

d) Time switch control to bend the missile over toward the target after it was launched vertically

After elaborate prepns requiring much time, personnel and equipment, the V-2 was fired vertically from a metallic launcher. A few seconds after the V-2 was in the air, the time switch control caused the missile to bend gradually over in the direction of the target. After 1 min of flight, the motor was turned off leaving the missile at about a 45° angle and having a velocity of about 3400 mph. For the remainder of the flight, the V-2 followed the trajectory of a free body in space reaching a maximum height of about 50 miles before returning to the surface of the earth some 200 miles from the launching site with a velocity of approx 1800 mph causing the warhead and any remaining fuel to explode

C. Infrared (IR) Guidance System condisted essentially of a concave mirror directed toward a target emitting the infrared radiation. A rotating disc and a photocell connected by a wire to a mechanism regulated the right-left and up-down movements of the missile. A schematic view of such device is given on p 11 of Ref 1 and a general description on detection of the infrared is given in Chapter 5 of Ref 4. One of the IR homing devices was used on the Rheintochter, R-3 (Ref 3, p 229), while another IR device, called **Madrid,** was installed on the Enzian, E-4 missile (Ref 3, p 232)

D. Magnetic-Ballistic Guidance System, such as used in the V-1 (FZG-76) missile, called also a "Buzz Bomb", was simple, rugged and reasonably reliable. In this system the azimuth was controlled by a magnetic compass, the altitude by a barometric altimeter and the range by an air mileage measuring unit. Prior to launching the missile, the devices were manually set for the desired course, altitude and range. The compass was linked to the directional gyroscope, whereas the altimeter acted directly on the elevator control system. All of the controls and amplifiers were pneumatic and the high-pressure air was stored in two tanks. When the predetermined range was reached in flight, the warhead was actuated and armed. The controls were then locked causing the missile to dive. The accuracy of the terminal portion of the flight depended upon the ballistics of the missile. (Ref 4, pp 35–36, 327–8 & 335–7). For more information on guidance systems for V-1 see Ref 3, pp 207–9. Some V-1 bombs were equipped with a one-tube radio transmitter for enabling the launching crew to follow the flights with direction finding equipment in order to obtn plotting and wind data (Ref 3, p 209)

E. Radar Guidance System or Radio Detection and Guidance System was not sufficiently developed to be used on a wide scale. Radar tracking of the target was used for guiding the Wasserfall and Rheintochter missiles (Ref 3, p 227 and Ref 4, p 41)

F. Radio Controlled Guidance Systems con-

sisted essentially of a radio receiver (located in a missile), a missile tracker and a radio transmitter (located near a missile launcher) for conveying the command to the receiver. This system was used in the majority of German guided missiles

Hs 293A was the first German radio controlled bomb. It was made in 1940 by Henschel, by equipping with radio control devices, the non-guided glide bomb designed in 1939 by the Gustav Schwartz Propellerwerke (Ref 2, p 202)

The following German radio controlled systems are listed or briefly described in Refs 3 & 4:

a) **Burgund** system consisted of an optical (visual) missile tracker, **Knüppel,** with a joy stick control, a radio receiver, **Strassburg** and a transmitter, **Kehl.** The Strassburg-Kehl combination was used in the PX-1400 glider bomb, Schmetterling (Hs 117) rocket, Wasserfall (C-2) rocket and Great Enzian rocket (Ref 3, pp 215–16, 223 & 232 and Ref 4, pp 38–43)

As a substitute for the Strassburg-Kehl command link, the **Kran-Brigg** system was developed late in WWII (Ref 4, p 41)

b) **Elsass** system was similar in operation to the Burgund's, except that radar tracking of the target replaced the optical tracking. It was proposed for use with the Rheintochter 3 and some other missiles (Ref 3, p 227 and Ref 4, p 41)

c) **Sonne** radial guidance system was based on the method which a navigator of a ship uses to determine its position by plotting the reverse bearings obtd from the radio transmitters of two known locations. The device Sonne was more complicated than the systems used in ship navigation. A brief description of the principles applied in the Sonne is given in Ref 4, p 595

d) **Friesicke & Höptner** radio receiver, first mounted on a Hs 293 missile proved to be too heavy and complicated for use. It was replaced by the **Staru** radio receiver (Ref 3, p 199)

e) **Stuttgart** radio telemetering system was tested on the Feuerlilie F-55 missile (Ref 3, p 226)

f) **Strassfurt** radio control system designed by the Rundfunk Co was planned to be used in the Enzian missiles (Ref 3, p 232)

g) **Kögge** radio control system designed by the Telefunken Co was intended for use in Enzian missiles (Ref 3, p 232)

G. Wire Controlled Guidance Systems. Owing to the fact that radio command guidance systems were susceptible to electronic countermeasures (jamming), a control by wires was developed. The system was installed in the X-4 air-to-air missile and was planned to be installed on the X-7 surface-to-air missile and some Henschel missiles (Ref 3, pp 205 & 216–16 and Ref 4, p 41). The wire links system was effective over short distances without fear of enemy countermeasures (See also Ref 3, p 217)

Since the wire method of control restricted the maneuverability of launching planes and required that they remain in the vicinity of missiles, thus exposing themselves to the weapons of enemy's bombers, the wire control method was replaced in the latter model of the X-4 by an acoustic homing device Kranich. With the latter device the parent plane could execute an evasive maneuver the moment the missile was launched and then withdraw itself beyond the range of enemy bombers' weapons (Ref 3, p 216)

The following varieties of wire command links systems are briefly described in Ref 4, pp 41–2:

a) **Dortmund-Duisburg** system consisted of an optical joy-stick control unit, a transmitting unit, two spools with wires (as described above) and a receiver located in the fuselage of X-4. The transmitting equipment consisted of an oscillator (operated by pulses from the joy-stick control) and an audio power amplifier which transmitted two audio-frequency signals thru wires to the receiving set in the missile. The audio signals were demodulated by the receiver to operate two polarized relays, one for pitch and another for yaw control

b) **Deren-Detmold** wire command link was a simple direct-current device which employed no vacuum tubes. The signals were transmitted to the receiver, which consisted of three relays. The 1st relay was sensitive to the polarity of the direct current signals (pitch control), the 2nd relay was sensitive to the amplitude of the signal (yaw control)

and the 3rd served to disconnect the other two when the transmitting wires were broken. In this case, the missile continued to follow the course of the last command received. The wires were the same as with the Dortmund-Duisburg system except that insulation was not removed, since it was essential in this system to keep the resistance of wires constant
Note: In all wire control systems, the fall of wire to the earth proved to be a nuisance and a hazard
Refs for Guidance Systems: 1) L.E. Simon, "German Research in World War II", J. Wiley, NY (1947), p 11 2) K.W. Gatland, "Development of the Guided Missile", "Flight Publication", London (1952), pp 13–16 3) Anon, "German Explosive Ordnance", Dept of the Army Technical Manual, **TM9–1985–2** (1953), Washington, DC 4) A.S. Locke et al, "Guidance", VanNostrand, NY (1955) 5) R. Guiliano, MAF **32**, 539–88 (1958) (Abbreviated translation from "Rivista Militare", Sept 1955) 6) B.T. Fedoroff et al, "Dictionary of Explosives, Ammunition and Weapons" (German Section), PATR **2510** (1958), Dover, NJ, pp Ger 83–85 7) M. Hobbs, "Basics of Missile Guidance and Space Technique", J.P. Rider, NY (1959)
Note: More recent refs on "Guidance" are given under "GUIDED MISSILE", Addnl Refs

Guided Aircraft Missile (GAM). A guided middile designed for launching from an aircraft in flight
Ref: GlossaryOrdn (1959), 141-L

Guided Aircraft Rocket (GAR). A rocket-powered guided missile designed to be launched from an aircraft in flight
Ref: GlossaryOrdn (1959), 141-L

Guided Bomb. An aerial bomb guided during its drop in range or azimuth, or in both
Ref: GlossaryOrdn (1959), 43-L (Bomb, guided)

GUIDED MISSILES. (Gesteuertes Geschoss in Ger; Projectile guidé in Fr; Oopravliayemyi snariad in Russ; Missile telecomandato in Ital; Proyectil guiado, in Ital)
History. Historically, guided missiles, also called *directed missiles*, were first used by Germans during WWII. They ranged from simple standard type aerial bombs, fitted with special equipment which permitted them to be directed thru a portion of their flight path, to V-1 and V-2 missiles. Americans, British and Russians used also during WWII some guided missiles, but none of them ever approached the qualities of German V-1 & V-2. After capitulation of Germany, all her enemies rushed to learn as many secrets on guided missiles as possible. Fortunately, "lion's share" came to Americans and Britishers, because Germans fearing Russian invasion transferred, near the end of the war, most of their equipment, including everything from Peenemünde, from east to west, and key personnel, such as Drs Werner von Braun and W. Dornberger. Since then, the Americans and British improved on German models and created their own. They are better equipped now than Germans were before and during WWII
General Description of Guided Missile. An unmanned, self-propelled vehicle capable of having its trajectory changed in flight to correct its path for errors or target motion. Generally, guided missiles are used for military purposes and travel above the earth's surface. The term "guided" is reserved for aerodynamic missiles and does not include naval torpedoes, which maintain a preset course, or ballistic missiles (See Vol **2** of Encycl, p B6-L & R), which follow a ballistic trajectory after thrust is terminated
Classification. Missiles are commonly classified by their launch and target environments, as well as by popular names. Other methods of classifying guided missiles are by trajectory, speed (subsonic, sonic or supersonic), propulsion (air breathers, usually jets or rockets), guidance (command, inertial or homing), payload (such as nuclear, high explosive, or electronic jammer), and purpose (strategic or tactical, offensive or defensive)
Guidance Systems. (See also previous item: Guided System of a Missile). There are several types of guidance systems: (1) They carry, in the missile, devices which enable

them to compute where they are in relation to a precalculated course. This requires extremely accurate mechan ical and electronic devices built into the missile. Such a system is known as "**preset**" and is suited generally to the surface-to-surface missile where the target is fixed. 2) Another means is to control the missile from the ground by knowing precisely where it is at all times and predict where the target will be. With this missile, it is possible to send electronic commands by radio, or radar techniques to the missile to enable it to correct its course so as to hit the target. 3) A missile may be designed to fly automatically in an electromagnetic beam while the beam is pointed onthe target. This is the *beam-rider* method used principally in the field of anti-aircraft defense. 4) A *navigational* system causes the missile to orient itself automatically with respect to stars or other known reference points, such as radar stations, and steer itself along the prescribed course. 5) The *homing* system uses missile borne devices to identify the target by heat, light, radar, sound, or other means and correct its flight path to hit the target. Homing is applicable against nearby targets or for final corrections at the terminal end of a long trajectory. Some missiles, particularly the long range ones are guided by a *combination* of two or morr of these systeme

Applications and Advantages. Since WWII, all modern military forces have been reshaped to some degree around guided missiles. The type to receive the greatest emphasis during the early period of guided missile development was the surface-to-air missile **(SAM).** These are anti-aircraft missiles designed to combat the high performance aircraft and missiles. Within this category are mobile systems, naval ship installations, and fixed land based installations. Antiballistic missile SAM systems have an important place in the strategic nuclear force of Russia. Guided missiles offer marked advantages in air-to-air use over conventional guns and unguided rockets. The air-to-air guided missile permits effective attack from much greater ranges and nearly every angle than conventional weapons. The guided missile also provides increased accuracy of delivery while reducing the risk to the attacking aircraft. Surface-to-surface missiles supplement or, in many applications, entirely displace conventional artillery for attacking fixed or moving battlefield or naval targets. The missile can carry a much greater payload to a greater range and with greater accuracy

Guided Missiles, American. The following is a listing of various American missiles according to their type: *Air-to-air missiles* **(AAM)**: Falcon, Sidewinder/Sparrow; *Air-to-surface missiles* **(ASM)**: Bullpup, Hound Dog, Shrike, Walleye; *Surface-to-air missiles* **(SAM)**: Chaparral, Hawk, Nike-Hercules, Redeye, Talos/Terrier/Tartar; *Surface-to-surface missiles* **(SSM)**: Subroc, Shillelagh

There are: *Surface-to-underwater* (SUM); *Underwater-to-air* (UAM); *Underwater-to-surface* (USM); and *Underwater-to-underwater* (UUM) (Ref 6)

Missiles Developed by Other Countries. (AAM): Matra RS30 (France); Firestreak/Red Top (British); Alkali/Anat/Ash/Stoll/Awl (Russia); (ASM): Nord AS20/AS30 (France); Martel (British-French); Robot R604 (Sweden); Blue Steel (British); Kangaroo/Kipper/Kitchen/Kennel (Russia); (SAM): Bloodhound/Tiger cat (British); Thunderbird (British); Seacat/Seaslug (British); Guideline (SA-2) (Russia); Goa (SA-3) (Russia); Galosh (Russia); (SSM): Ikara (Australia); Malafon (France); Entac (France); SS-11/SS-12 (France); Cobra (German); KAM-3D (Japan), Bantam (Sweden); Mosquito (Swiss), Vigilant (British); Sagger/Swatter/Snapper and Styx (Russia); (See also "Ballistic Missiles" in Vol **2** of Encycl, p B86)

Refs: 1) F. Ross Jr, "Guided Missiles, Rockets and Torpedoes", Lothrop, Lee & Shepard Co, NY (1951), pp 66–167, pp 168–176 2) N.F. Toftoy, Ordn **38**, 592–4 (1954) (Rocket development) 3) Anon, Ordn **38**, 753–56 (1954) 4) A.S. Locke et al, "Guidance", VanNostrand, NY, Vol **1** (1955) 5) A. Zaehringer, "Rockets and Guided Missiles", Ordn **40**, 298–300 (1955) 5) W.H. Pickering, "Guided Missile Problems", Ordn **40**, 241–3 (1955) 6) GlossaryOrdn (1959), 141-L

Addnl Refs: A) B.H. Liddel Hart, "The

Revolution in Warfare", Yale University Press, New Haven (1947) (125pp)
B) J.C. Boyce, "New Weapons for Air Warfare", Little, Brown, Boston (1947) (353pp)
C) W.L. Clay, Ordn **32**, 154–56 (1947)
D) L. Ruiz, Ordn **32**, 165–6 (1947)
E) C.F. Green, Ordn **33**, 96–98 (1948)
F) Alessandro, Ejercito (Spain), 96 (1948)
G) E. Romat, "Les Robots Aériens", J de Gigord, Paris (1948)
H) A.R. Weyl, "Guided Missiles", Temple, London (1949)
I) N.A. Parson, Jr, Ordn **35**, 403–05 (1951)
J) J.T. Lewis, Ordn **36**, 842–45 (1952)
(Brief description of work done at Guided Missile Center, Fort Bliss, Texas
K) P.W. Powers, "The Aerodynamics of Guided Missiles", Combat Forces J, June 1952, pp 19–21, "Guided Missile Propulsion", July 1952, pp 35–7
L) K.W. Gatland, "Development of the Guided Missile", Philosophical Library, NY (1954)
M) S.M. Smolensky, Ordn **39**, 64–7 (1954) (Guided Missile Production)
N) J. Humphries, "Rockets and Guided Missiles", El Benn, Ltd (1956)
O) G. Merrill, "Principles of Guided Missiles Design", VanNostrand, NY, Vol **1** (1955), "Guidance"; Vol **2** (1956), "Aerodynamics, Propulsion, Structures"; Vol **3** (1956), "Operations Research, Armament, Launching"; Vol **4** (1960), "Systems Engineering, Range, Testing"; Vol **5** (1960), "Space Flight"; Vol **6** (1960), "Guided Missile Designer's Handbook"
P) N.A. Parson, Jr, "Guided Missiles in War and Peace", Harvard University Press, Cambridge, Mass (1956) (161pp)
Q) Anon, Ordn **41**, 249 (1956)
R) J.P. Jensen, "Rockets and Guided Missiles", Birk & Co, NY (1956)
S) Anon, KogakuGaho (Japan) **5**, No 13 (Nov 10, 1957) (List of Guided Missiles of the World) (A translation in English, OTIA No **2825**
T) E. Burgess, "Guided Weapons", MacMillan, NY (1957)
U) Eilene Gallaway, "Guided Missiles in Foreign Countries", USGovtPrtgOffice Pamphlet **UG630–U5**, Washington, DC (1957)
V) E. Bergaust, "Rockets and Missiles", Putnam, NY (1957)
W) J. Humphries, "Rockets and Guided Missiles", MacMillan, NY (1957), 231pp
X) Anon, "Guided Missiles Fundamentals", ATC 52–12, USAF Air Transport Command, Wash, Bureau of Naval Personnel
Z) Anon, "Guided Missiles Fundamentals", Dept of Air Force, AF Manual **52–31**, US-GovtPrtgOffice, Washington (Sept 1957) (575pp)
AA) U.D. Rose, Edit, "Die unheimliche Waffen. Atomraketen über uns. Lenkwaffen, Raketen-Geschosse, Atombomben", Schild-Verlag, München-Lochhausen (1957)
BB) Th. Benecke & A.W. Quick, "History of German Guided Missiles Development", Verlag-Appelhens & Co, Brunswick, Germany (1957)
CC) Martin Caiden, "Countdown for Tomorrow", Dutton, NY (1958)
DD) H.E. Newell Jr, "Guide to Rockets, Missiles and Satellites", McGraw-Hill, NY (1958)
EE) Barron Kemp, "What Makes Guided Missiles Tick", American Electronics Co, NY (1958)
FF) J.M. Chester, "An Introduction to Guided Missiles", Rand Corp **P-1327**, (1958)
GG) Staff, "Dept of the Air Force Guided Missiles, Operztions, Design and Theory", McGraw-Hill, NY (1958)
HH) Allen E. Puckett, "Guided Missile Engineering", McGraw-Hill (1959)
II) J. Clemow, "Short Range Guided Weapons", Temple Press Ltd, London (1961)
JJ) John W.R. Taylor, "Rockets and Missiles", Langacre Press Ltd, London (1962)
KK) Anon, "Encyclopedia of Science and Technology", McGraw-Hill, NY, Vol **6** (1971) pp 334–6

Guided Missile, Antitank. An A/T missile whose path is controlled by a combination of optical sighting and command signals from an automatic computer thru multiple wire command links. It may also contain an IR homing device for final range correction. It is designed to be launched from any type vehicle or ground emplacement. This excludes items whose trajectory cannot be altered in flight
Ref: GlossaryOrdn (1959), 141-L

Guided Missiles, German of WWII. Beginning in 1938 several successful guided missiles were developed in Germany, mostly at Peenemünde and Volkenrode (See also History, under GUIDED MISSILES)

One of the first successful missiles was *Rheinbote* (Rheinmessenger). It was described by Ross (Ref 2)

Other successful missiles are listed in Ref 5, p Ger 85 and briefly described in Refs indicated here. See also illustrations shown on p Ger 85 of Ref 5, reproduced here

Other successful guided missiles were:

a) **Schmetterling** (Butterfly), also known as the Hs-117 (Ref 2, p 35)

Note: Hs is an abbr for Henschel, the name of the builder

b) **Wasserfall** (Waterfall) (Ref 2, p 37)

c) **Rheintochter** (Daughter of the Rhein) series such as Rheintochter I, II & III (Ref 2, p 40)

d) **Enzian** (Gentian, a species of blue flower) series, ranging from E-1 to E-5 (Ref 2, p 43 & Ref 1, p 99)

e) **Feuerlilie** (Fire Lilly) series, of which the Hecht (pike) was one of the first successful. T-Stoff and Z-Stoff were used in it. The Hecht was succeeded by the Feuerlilie F-25. The last of the series was the F-55, used only for research (Ref 2, pp 45–47, Ref 1, pp 95–96)

f) **Bachem BP-20 Natter** (Viper) (Ref 2, p 47)

g) **Ruhrstahl** (Steel of the Ruhr) series ran from X-1 to X-7, of which the X-4 was the most important (Ref 2, p 50 & Ref 1, pp 90–2)

h) **Hs** (Henschel, the name of builder) series, including the previously mentioned Hs-117 (Schmetterling), as well as Hs-117H, Hs-293, Hs-294, Hs-295, Hs-296 & Hs-298 (Ref 2, pp 52–54 & 56–60, Ref 1, pp 92–3)

i) **Fritz X** (FX-1400), a glide bomb (Ref 2, p 55)

j) **Beethoven Apparatus** – an odd looking guided missile (Ref 2, pp 61–62)

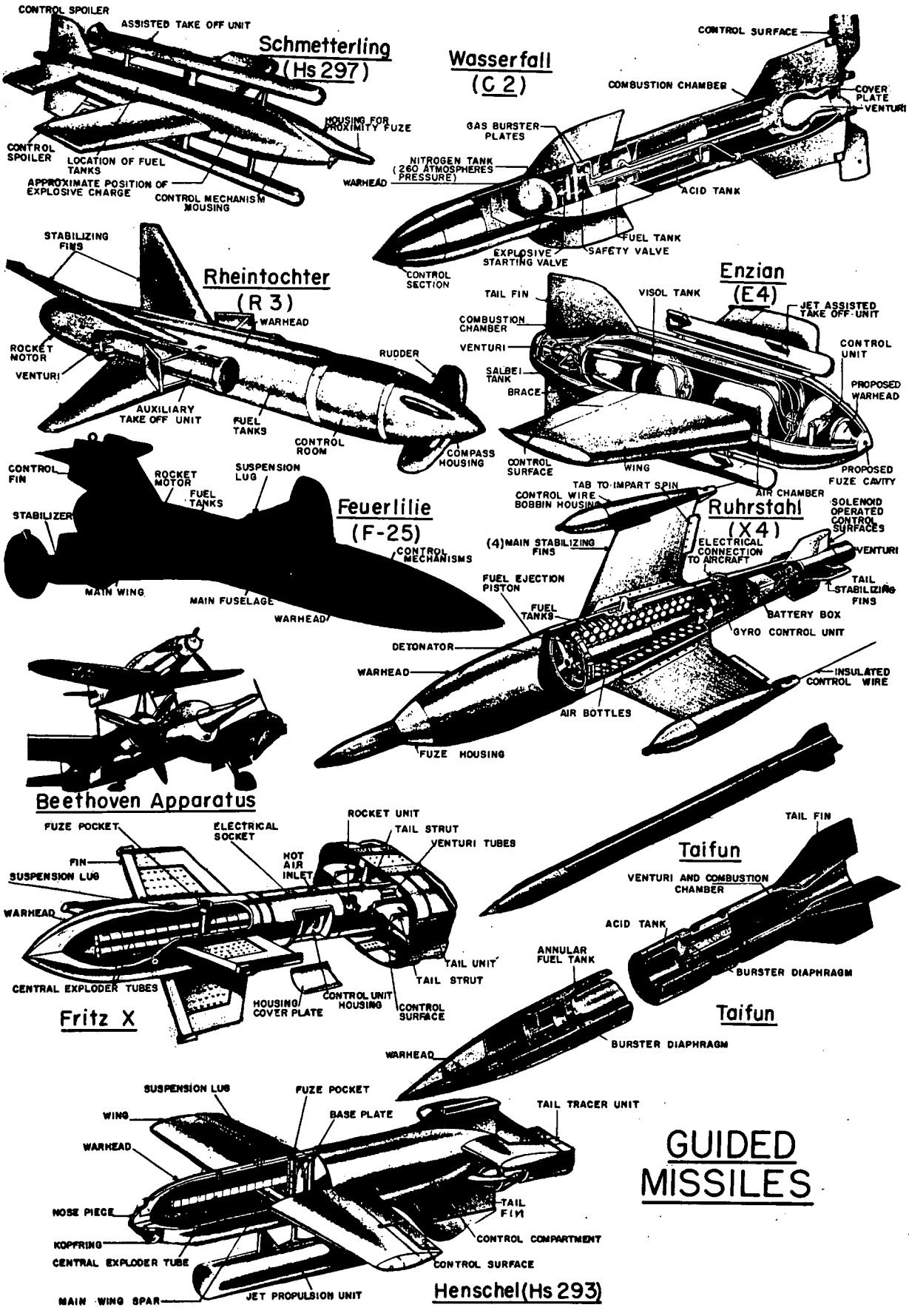
k) **BV-246** (Ref 2, p 63)

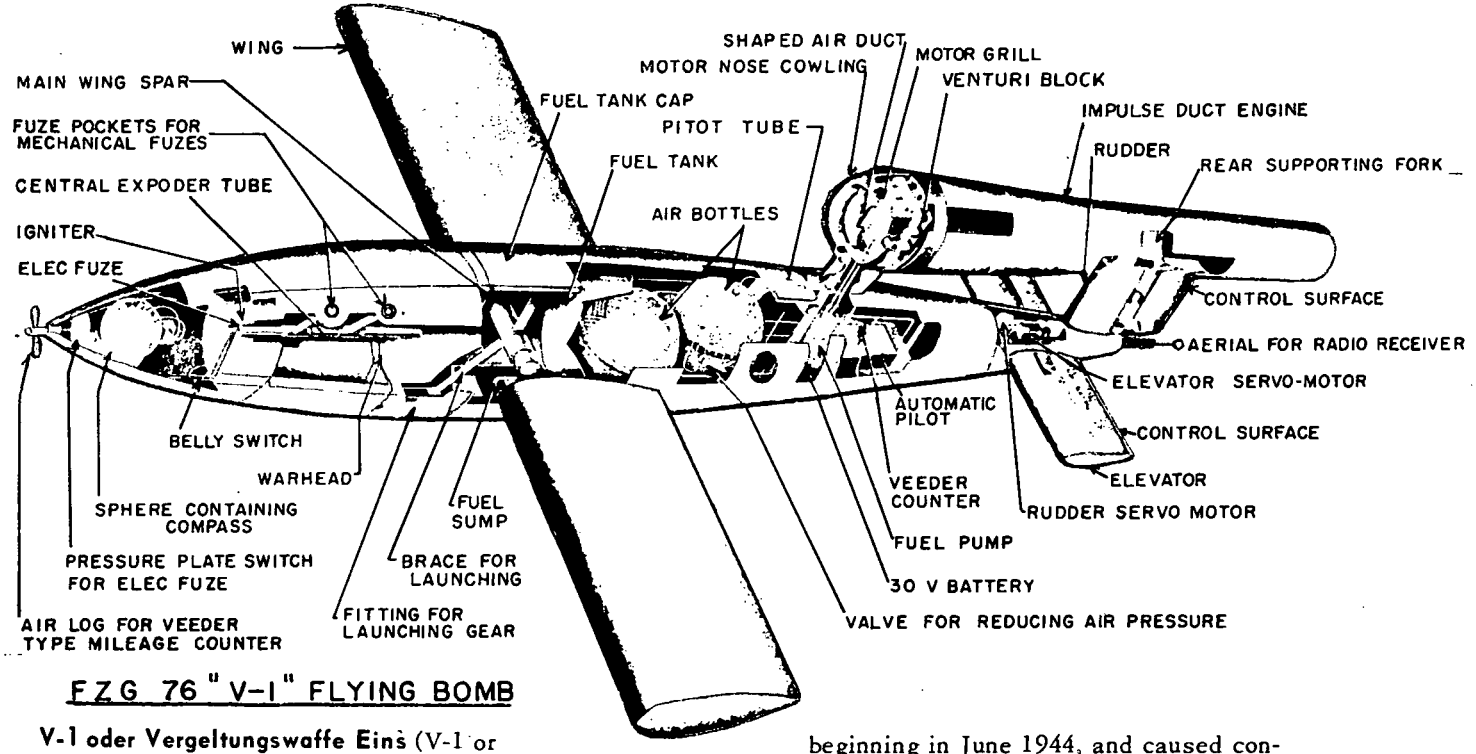
l) **V-2**, is briefly described separately under V-2 and below. It could be launched as a guided missile (Ref 5, p Ger 213)

m) **Antipodol Bomber** (Ref 3, pp 57–58)

n) **Taifun**, a biliquid rocket (Ref 4, p 223)

Refs: 1) A. Ducrocq, "Les Armes Secrètes Allemendes", Berger-Levrault, Paris (1947), 90–9 2) F. Ross Jr, "Guided Missiles, Rockets and Torpedoes", Lothrop, Lee & Shepard, NY (1951), 14–60 3) K.W. Gatland, "Development of the Guided Missile", "Flight" Publication, London (1952), 2–19, 47 & 49–59 4) Anon, TM **9–1985–2** (1953), 195–233 5) B.T. Fedoroff, et al, PATR **2510** (1958), pp Ger 82 & 85 (Guided Missiles) & 213 (V-2)


CONTROL SPOILER
ASSISTED TAKE OFF UNIT
Schmetterling
(Hs 297)
HOUSING FOR PROXIMITY FUZE
CONTROL SPOILER
LOCATION OF FUEL TANKS
APPROXIMATE POSITION OF EXPLOSIVE CHARGE
CONTROL MECHANISM HOUSING
Wasserfall
(C 2)
CONTROL SURFACE
COMBUSTION CHAMBER
COVER PLATE
VENTURI
GAS BURSTER PLATES
NITROGEN TANK (260 ATMOSPHERES PRESSURE)
WARHEAD
ACID TANK
FUEL TANK
SAFETY VALVE
EXPLOSIVE STARTING VALVE
CONTROL SECTION
STABILIZING FINS
Rheintochter
(R 3)
WARHEAD
ROCKET MOTOR
VENTURI
RUDDER
AUXILIARY TAKE OFF UNIT
FUEL TANKS
CONTROL ROOM
COMPASS HOUSING
Enzian
(E4)
TAIL FIN
VISOL TANK
JET ASSISTED TAKE OFF UNIT
COMBUSTION CHAMBER
VENTURI
CONTROL UNIT
SALBEI TANK
BRACE
PROPOSED WARHEAD
CONTROL SURFACE
WING
PROPOSED FUZE CAVITY
AIR CHAMBER
CONTROL FIN
ROCKET MOTOR
FUEL TANKS
SUSPENSION LUG
STABILIZER
Feuerlilie
(F-25)
CONTROL MECHANISMS
MAIN WING
MAIN FUSELAGE
WARHEAD
TAB TO IMPART SPIN
CONTROL WIRE BOBBIN HOUSING
Ruhrstahl
(X4)
SOLENOID OPERATED CONTROL SURFACES
(4) MAIN STABILIZING FINS
ELECTRICAL CONNECTION TO AIRCRAFT
VENTURI
FUEL EJECTION PISTON
TAIL STABILIZING FINS
FUEL TANKS
BATTERY BOX
GYRO CONTROL UNIT
DETONATOR
WARHEAD
INSULATED CONTROL WIRE
AIR BOTTLES
FUZE HOUSING
Beethoven Apparatus
FUZE POCKET
ELECTRICAL SOCKET
ROCKET UNIT
TAIL STRUT
VENTURI TUBES
FIN
HOT AIR INLET
SUSPENSION LUG
WARHEAD
TAIL UNIT
TAIL STRUT
CENTRAL EXPLODER TUBES
HOUSING/ COVER PLATE
CONTROL UNIT HOUSING
CONTROL SURFACE
Fritz X
Taifun
TAIL FIN
VENTURI AND COMBUSTION CHAMBER
ACID TANK
BURSTER DIAPHRAGM
ANNULAR FUEL TANK
Taifun
BURSTER DIAPHRAGM
WARHEAD
SUSPENSION LUG
FUZE POCKET
TAIL TRACER UNIT
WING
BASE PLATE
WARHEAD
NOSE PIECE
TAIL FIN
KOPFRING
CONTROL COMPARTMENT
CENTRAL EXPLODER TUBE
CONTROL SURFACE
MAIN WING SPAR
JET PROPULSION UNIT
Henschel (Hs 293)
GUIDED MISSILES



FZG 76 "V-1" FLYING BOMB

V-1 oder Vergeltungswaffe Eins (V-1 or Revenge Weapon One). The official German designation was FZG-76 and the British name **Buzz Bomb.** V-1 was a pilotless plane (winged rocket) which could fly at a speed of 500–560 mph at a height between 2000 & 3000 feet and to a distance of 220 miles. It could be launched from a catapult or released from a piloted plane. The body of the V-1 rocket was cylindrical in shape, tapering toward the nose; diam 2ft, 7 inches and total length 21ft, 5 inches. Fully loaded it weighed 4750 lbs. It was propelled by a pulse-jet engine using 150 gallons of gasoline for fuel and compressed air as the oxidizer. The warhead contd some newly developed explosives which could withstand high temps. These rockets were fired against England, beginning in June 1944, and caused considerable damage

Refs: A. Ducrocq, "Les Armes Secrètes Allemandes", Berger-Levrault, Paris (1947), p 35 2) F. Ross, Jr, "Guided Missiles, Rockets and Torpedoes", Lothrop, Lee, Shephard, NY (1951), pp 14–20 3) K.W. Gatland, "Development of the Guided Missile", "Flight" Publication, London (1952) 4) Anon, "German Explosive Ordnance", TM 9-1985-2 (1953), pp 205–10 5) W. Dornberger, "V-2", The Viking Press, NY (1954), p 93–98 6) A.S. Locke et al, "Guidance", Van Nostrand, NY (1955), pp 34–5, 56–7, 71 & 76 (Book 1 of the "Principles of Guided Missile Design", edited by Grayson Merrill)

Guided Missile, German V-2 oder Vergeltungswaffe Zwei (V-2 or Revenge Weapon Two). The official German designation was A-4. V-2 was a rocket provided with 4 stabilizing fins and control radio. It could fly with a speed up to 3600 mph to a distance up to 220 miles and at altitudes up to 50–60 miles. The body of the rocket was cylindrical in shape with a nose tapering to a sharp point. The largest diameter was about 5 ft and the overall length 46 ft. Fully loaded it weighed about 14 tons, which included 9 tons of fuel supply and about 1 ton of special explosive that could withstand high temps in the warhead. The first of these rockets was fired against England in Sept 1944. A total of 115 V-2 rockets were fired up to April 2, 1945, and they caused considerable damage especially in London and vicinity

Table 62 gives some additional information on V-2, as taken from the book of Gatland (Ref 3, p XVII)

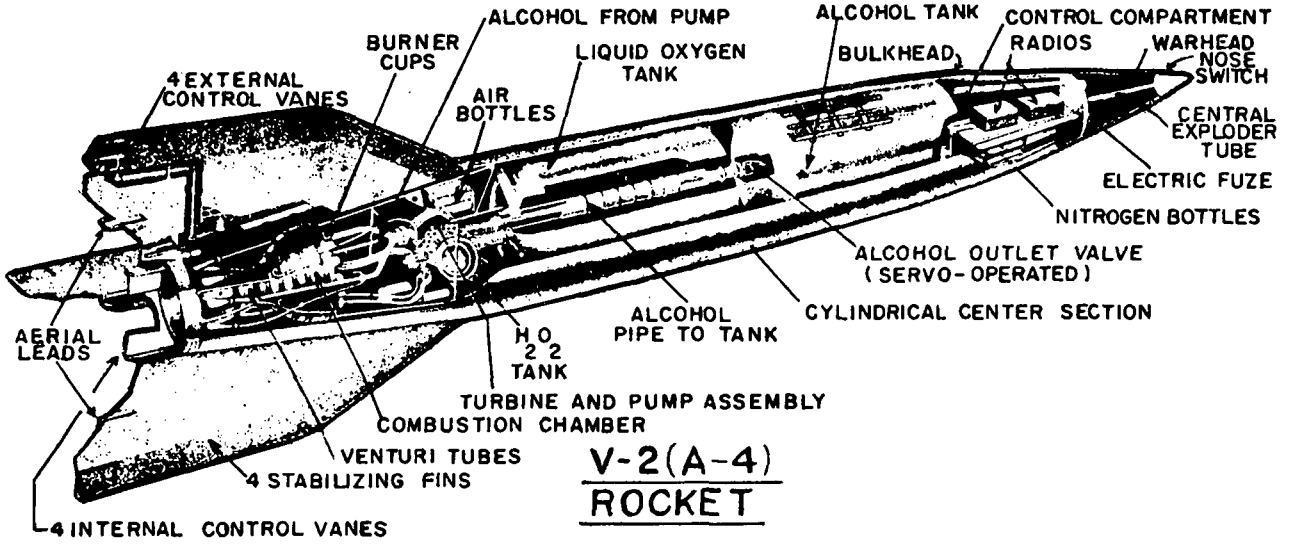
TABLE 62

Characteristics of V2	Metric	U S
Length	14 m	46'
Diameter of body	1.65 m	5'5"
Take-off weight	12,900 kg	28,440lb
Payload	1000 kg	2205lb
High explosive carried	750 kg	1654lb
Alcohol (Contg 25% water)	3965 kg	8740lb
Liquid oxygen	4970 kg	10,957lb
Thrust at take-off	25,000 kg	55,100lb
Thrust gain near Brennschluss	4200 kg	13,230lb
Fuel consumption per second	127 kg	280lb
Alcohol / oxygen ratio in mixture	0.81	
Maximum burning time	65 sec	
Temp in motor	~2700° C	~4890°F
Pressure in motor	15.45 atm	227 psi
Nozzle expansion ratio	15.45:0.85	
Exhaust velocity	2050 m/sec	6725 ft/sec

Refs: Same as under Guided Missiles, German of WWII and W. Domberger, "V-2", The Viking Press, NY (1954), 93–8

Note: The description given here is from PATR **2510** (1958), p 213

Note: Its predecessor **V-1** (Vergeltungswaffe Eins), known as "Buzz Bomb" was not guided. It is described in PATR **2510** (1958), p Ger 213-L with Fig on p Ger 214

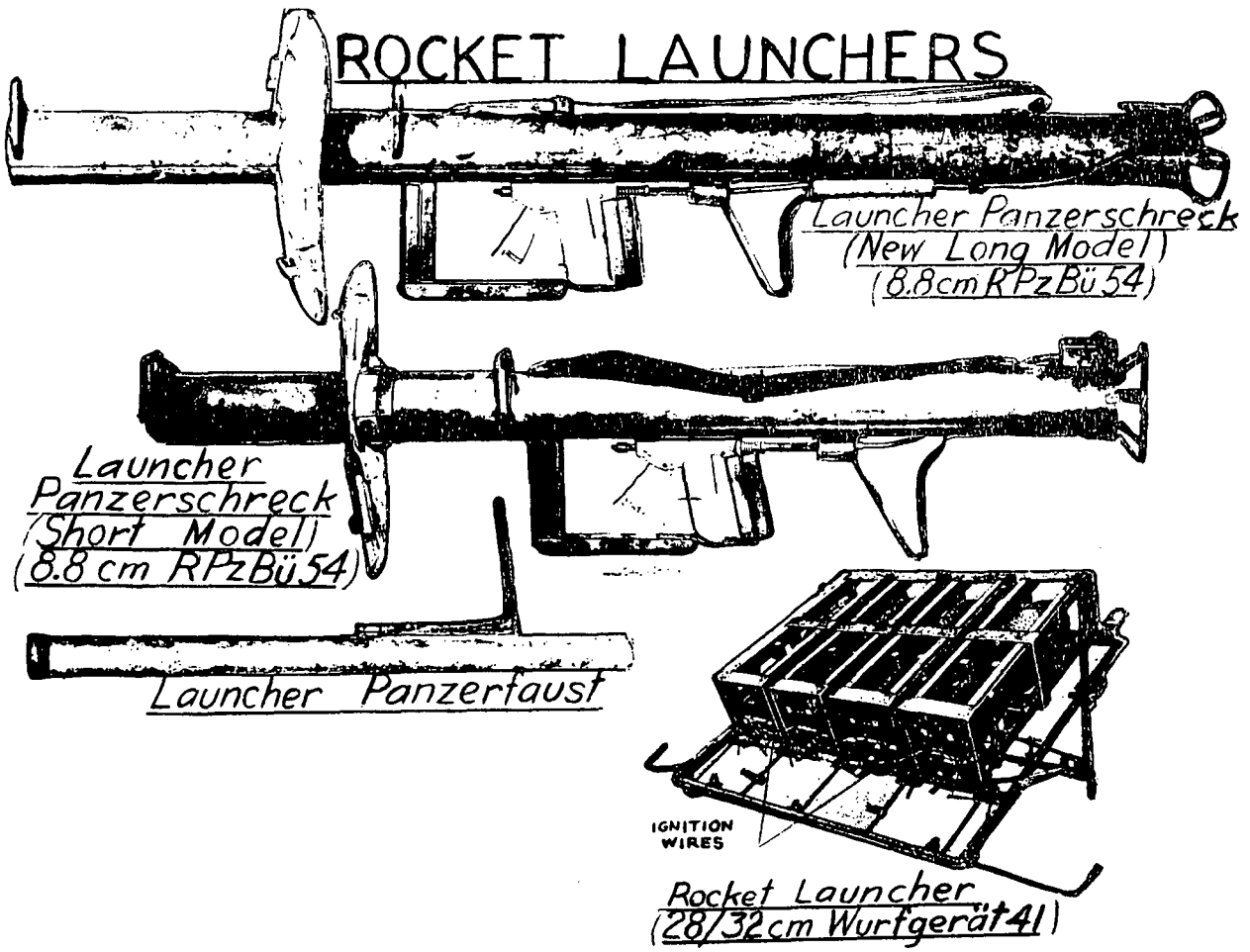


Guided Missile Launcher. A device or installation from which a self-propelled missile is launched. It usually incorporates a rail, tube, wooden or steel frames, etc for giving the missile initial guidance. Some of the launchers are the same as used for launching rockets. Germans developed and used during WWII many types of launchers and nearly all of them are described in PATR **2510**(1958), p Ger 164 under Rocket Launcher or Projector with numerous illustrations on p Ger 165. Some of them are shown in the Figs included here

For more info on guided missile launchers see Ref 2 and books on guided missiles and under Launchers

Refs: 1) GlossaryOrdn (1959), 168-L 2) Anon, "Principles of Artillery Weapons", **TM9-3305-1** (1956), pp 12 & 171-72 3) US Military Specification MIL-L-11992 (Ord) 4) Vol **2** of Encycl PATR **2700**(1962), p C29-L (Guided Missile Launchers) and C28-R (Rocket Launcher)

Note: Illustrations of NIKE and CORPORAL launchers used by the US Armed Forces are given on pp 173-74 of Ref 2



GUM is a general term for thick, mucilagenous excretions from various plants. It consists of complex carbohydrates which yield sugars on hydrolysis. The gums either dissolve or swell in water and may be pptd by alcohol

Gums may be divided into 3 classes: a) *Arabin* type, completely sol in w, such as gum arabic; b) *Bassorin* type, such as gum dragon, sl sol in w and c) *Cerasin* type, such as cherry gum, swelling in w

Some of the gums have been used in Dynamites to increase their plasticity (Ref 1, p 406), but their principle use in the expl industry is as binding agents in priming and initiating compns. Being carbohydrates, they function to a certain extent as combustible materials

Gum Arabic or Acacia Gum (Gomme, in Fr). A white powder obtd by drying the gummy exudation from the bark, stems and branches of the Acacia Senegal trees (or other acacia species). It is completely sol in w, forming a glue which is fairly strong. It has been used in some expls, such as *Grenite*, and in some priming mixts. It has also been used in some Pyrotechnic compns, as the following colored smokes: K perchlorate 25, antimony sulfide 20, dye (such as rhodamine red, methylene blue or malachite green) 50 and gum arabic 5

Due to the hygr of gum arabic, it has been replaced in some mixts by linseed oil and, more recently, by *Parlon*, which is Hercules Powder Co chlorinated rubber, supplied as a white granular powder intended for use in corrosive resistance paints as a film former (Ref 7)

Requirements of the US Armed Forces for gum arabic are covered by Spec JJJ-G-821, and are as follows:

1) *Insoluble residue* – max 1.0%
2) *Total ash* – max 4.0
3) *Acid-insoluble ash* – max 0.5
4) *Moisture* – max 15.0
5) *Tannin-bearing gums* – none
6) *Starch and dextrin* – none
7) *Identification* When 0.2ml of 4% Pb subacetate is added to 10ml of 2% aqueous soln of gum arabic, a flocculent or curdy ppt shall form immediately
8) *Solubility.* When 35g of the sample is added to 100ml water and allowed to stand, with occasional shaking for 18–24 hours, the resulting liquid shall be free-flowing, uniform in appearance and without any indication of ropiness
9) *Reduction of Fehling's solution.* Not more than a trace of CuO shall be formed when the material is tested with Fehling's solution
10) *Acidity* – Inorganic, none; Organic, max 0.40% as acetic acid

Gum, Green. This is not really a gum but finely powdered coconut shell. It is not sol in w (Ref 4, p 4)

Gum Dragon or Gum Tragacanth. A gummy exudation from the stems of trees, such as Astragalus gummifer etc, mostly native to Asia Minor. It is sl sol in w and is used as a binding agent in some Priming Compositions in lieu of gum arabic etc (Ref 1, p 406, Ref 2, p 455 and Ref 5, p 1154-R). Requirements of the US Armed Forces are covered by Spec JAN-G-96A, and are as follows:

1) *Color* – white to light tan (by visual observation)
2) *Form* – leaf or flake-like (by visual observation)
3) *Odor* – none
4) *Chlorine as chlorides* – none
5) *Acidity* – Inorganic, none; Organic, max 0.40%
6) *Foreign gums* – none
7) *Indian gums* – none
8) *Ash* – max 3.5%

Gum, Guar is described here separately as **Guar Gum**

Gum Kauri (Copal Resin). This is not really a gum because it is not sol in w and does not swell in it. It is a fossil resin obtd from New Zealand. It is yel to yel-brn, insol in w and the usual solvents. It has been used in some pyrotechnic compns (Ref 3, p 466-L & Ref 4, p 4)

Gum Lac or **Shellac.** It is obtd from a resin secreted by the insect Laccifer lacca and deposited on the twigs of various species of trees in India, Siam and Indo-China. The collected material is dried, ground and washed to remove the adherent red dye. Then the material is melted, strained and flaked, similarily as in procedures described in this Vol, p F44-L, under Flaking. This gives orange shellac of commerce. White shellac is obtd by bleaching orn shellac with Na hypochlorite. Both are insol in w, but sol in alc. Their extensive list of uses includes: paints, varnishes, dielectric compns, sealing wax and match heads (Ref 3, 768-L & Ref 5, 1018-L)

Gum-resins (Oleoresins). A group of aromatic exudations of plants which consist of a mixt of various substances (as essential oils) with the gum. The following are official (USP): ammoniac, assafeteda, gamboge, myrrh and scammony [Ref 3, p 392-L & Ref 5, p 327-R (Oleoresins)]

Gum Sandarac or **Juniper Gum.** The resinous substance which exudes from *Callitris quadrivalvis*, found in Morocco; yel, brittle, amorph substance; sol in alc, eth, acet, chlf & other solvents, insol in w, benz & light petr hydrocarbons. Used as incense, varnishes, lacquers and probably suitable as binder in expl compns [Ref 3, p 755-L (Sandarac) & Ref 5, p 999-L (Sandarac Gum)]

Gum-sugar (Arabinose), $C_5H_{10}O_5$, is a syrupy concd sugar soln in glycerol which does not crystallize. It was used in Germany in some Dynamites when it was required to render them plastic without the addn of a large amount of NG–NC gel. It might be said that gum-sugar saved or "stretched" NG gelatins [Ref 5, p 103-R (Arabinose)] [See also under "Extending (Stretching or Lengthening) Gelatin-Dynamites in this Vol, p E496]

Gum-tree (Red gum, Sweet gum). Gum obtd from a tree grown in the swamps of the Mississippi river and from the Kangaroo islands near Australia. It has been used as a binding agent in some pyrotechnic compns, eg in the following "Simulated Booby Trap" illuminating compn: Red gum 13 to 15, dextrin 5.5 to 6.5 & $KClO_4$ 78–82% (Refs 1, 3 & 4)

Refs: 1) Naoúm, NG (1928), p 406 2) Davis (1943), 455 (Gum arabic and Gum tragacanth) 3) Hackh's Dict (1944), 392-L (Gums); 392-L (Gum-resins); 392-R (Gum-tree); 466-L (Kauri Gum) 4) G.W. Weingart, "Pyrotechnics", ChemPubgCo, Brooklyn (1947), 4–5 & 194–95 5) CondChemDict (1961), 334-R (Gums); 103 (Arabic Gum); 103 (Arabinose); 851-R (Parlon); 1154-R (Tragacanth Gum) 6) Kirk & Othmer, Vol **10** (1966), pp 741–54 (Gums, natural) 7) CondChemDict (1971), 75 (Arabic gum), 429 (Gum, natural), 659 (Parlon) 8) US Specification JAN-G-276 (Gum, ester); MIL-G-96A (Gum, Tragacanth) (For use in ammo)

Gum Incendiaries, such as 8% rubber soln in benzene, proved to be (when used in incendiary bombs) about ½ as effective on the wt basis as magnesium bombs. Being a sticky material, gum incendiary proved to be particularly suitable for use in scatter bombs. This material was about 5 times as effective as Thermite or SDO-sodium nitrate incendiaries
Ref: L.F. Fieser, OSRD **173** (1941)

Gummidynamit. A German rubber-like, elastic mass obtd by dissolving Collod Cotton in NG. Its compn is similar to Sprenggelatine (Blasting Gelatin)
Ref: PATR **2510** (1958), p Ger 83

GuN. Abbrn for Guanidine Nitrate

GUN. There is great confusion in definition of this term

Greener (Ref 1, p 565) defined it as "a thermodynamic machine by which the potential energy of the explosive is converted into the kinetic energy of the projectile". Although he does not describe in his book any artillery pieces, the meaning of his definition is that

any weapon which uses an explosive for propelling a projectile is a gun. This corresponds to definition given in Glossary of Ordn (Ref 7), in which it is said that gun, in general, is a piece of ordnance consisting essentially of a tube or barrel, for throwing projectiles by force, usually the force of an explosive, but sometimes that of compressed gas, spring, etc. This general term embraces such weapons as are sometimes specifically designated as: gun, howitzer, mortar, cannon, firearm, rifle, shotgun, carbine, pistol and revolver. To these should be added recoilless gun, machine gun, submachine gun, rocket launcher and guided missile launcher. Specifically, a gun can be defined as a long-range artillery piece with a relatively long barrel (usually longer than 30 calibers), with relatively high initial velocity, high pressure, and capable of being fired at a low angle of elevation, say under 20°

The first of these definitions (general) is contradictory to definition given in TM9–3305–1 (Ref 6), which is described in Ref 8, p C26-R. It defines *a gun, as a cannon* and then it is said that the term "gun" is also applied to all firearms. More confusion was added by Ohart (Ref 4, p 3) who said: "A cannon is any large weapon too heavy to be carried by hand. Guns, howitzers and mortars (except trench mortars) are types of cannon"

If definition of gun given in Ref 7 is correct, then the definitions of other weapons should be reworded as:

a) *Howitzer* is a *gun* with a length of barrel shorter, chamber pressure & muzzle velocity lower, range shorter and angle of fire (up to 65°) higher than those of guns (Ref 8, p C27-L)

b) *Mortar* is a *gun* with a barrel shorter, muzzle velocity lower, angle of fire higher (up to 85°) and range shorter than those of howitzers (Ref 8, p C27-R)

c) *Recoilless Rifle* is a *gun* which permits the discharge of projectiles with no rearward movement of barrel or mount (recoil) (Ref 8, p C28-R)

Other definitions given in Ref 8 include *trench mortar* (p C28-L), *rocket launcher* (p C28-R) and *guided missile launcher* (p C29-L)

The following US guns of WWII are listed in Ref 8, p C27-L: *field, aircraft, anti-aircraft, tank, railway* & *seacoast* and Naval. There were also *antitank* (A/T) guns

The following artillery weapons are now used by the US Armed Forces:

Weapon	Use
37-mm Automatic Gun, M1A2	Antiaircraft
40-mm Gun Cannons, M1, M1A1, M2, M2A1 & Mk 1 (Navy)	Antiaircraft
57-mm Recoilless Rifles, M18, M18A1 & T15E16	Direct Fire
60-mm Mortars, M2 & M19	Ground Fire
75-mm Gun Cannon, M3	Tank Weapon
75-mm Gun Cannon, M35	Antiaircraft
75-mm Pack Howitzer Cannon, M1A1	Field Artillery
75-mm Recoilless Rifles, M20 & T21E12	Direct Fire
76-mm Gun Cannons, M32 & M48	Antitank
81-mm Mortars, M1 & M29	Ground Fire
90-mm Gun Cannons, M1 & M2	Antiaircraft
90-mm Gun Cannons, M36, M41 & M54	Antitank
90-mm Recoilless Rifle, M67	Direct Fire
105-mm Gun Cannon, M68	Antitank
105-mm Howitzer Cannons, M2, M2A2, M4, M4A1 & M49	Field Artillery
105-mm Recoilless Rifles, M27 & M27A1	Antitank
106-mm Recoilless Rifles, M40A1 & M40A1C	Antitank
4.2-inch Mortar, M30	Ground Fire
120-mm Gun Cannons, M1A1, M1A2 & M1A3	Antiaircraft
120-mm Gun Cannon, M58	Antitank
152-mm Gun Cannon, M81	Aircraft Gun
155-mm Gun Cannons, M2 & M46	Field Artillery
155-mm Howitzer Cannons, M1, M1A1, M45 & M126	Field Artillery
165-mm Gun Cannon, XM135	Field Artillery
175-mm Gun Cannon, M113	Field Artillery
8-inch Howitzers, M2 & M2A1	Field Artillery
280-mm Gun Cannon, M66	Field Artillery

Ohart (Ref 4, p 173) tabulates the calibers of US artillery weapons used by the Army during WWII. The categories listed are mobile, railway & seacoast, tank armament, antiaircraft, and aircraft armament

Note: Accdg to Staff, Ordn **38**, 607 (1954), a gun was developed which is capable of firing atomic shells. This gun is a caliber 280mm weapon and can hurl projectiles with greater accuracy than any other artillery piece. It can move along a highway at 35 miles/hr across country, can be emplaced in mins, and can be fired around the compass. The gun is so delicately balanced that one man can elevate or depress it by hand should the power mechanism fail. This has been a most dramatic advance in a program to build firepower for defense noted the US Army Secretary

The Germans developed before and during WWII more new types of guns than all other Nations (Austria, Belgium, Czechoslovakia, France, Gt Britain, Italy, Japan, Poland, Russia, Spain, United States of America and Yugoslavia). A practically complete list of their guns and of other weapons (including numerous illustrations), beginning with 2-cm cannons and ending with 80-cm Gustav Geschütz, used at the siege of Sevastopol' is given in PATR **2510** (1958), pp Ger 237 to Ger 262, incl Als

Note: Germany listed calibers of their guns in cm instead of mm

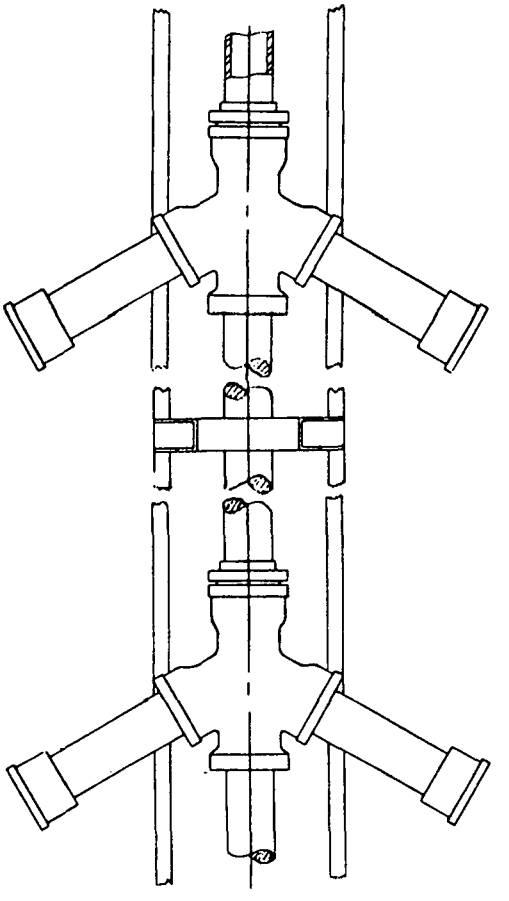
One of their best known guns was invented during WWI. It was *Paris Gun* (Pariskanone oder Ferngeschütz). It existed in several calibers and lengths and was described in Vol **2** of Encycl, p B113-R, under Big Bertha, although the name Big Bertha must be (accdg to W. Ley, CoastArtilleryJournal, Jan–Feb 1943, pp 14–15) referred to as 42cm, L/14 Howitzer and not as Paris Gun. Besides the Paris Gun (officially known as Kaiser Wilhelm Geschütz), several other big guns are listed on p B113-R and B114-L of Vol **2**

Although we cannot list all German guns, many of them conventional, it is necessary to list and describe two of them which have interesting construction, although they do not seem to be of practical use

One of them, known as *Hochdruckpumpe oder V-3* (High-Pressure Pump or Revenge Weapon-3) (V-3 stands for Vergeltungswaffe 3), also known as "HDP Supergun". It is described in PATR **2510** (1958), p Ger 90 with illustration shown below

The barrel, caliber 150mm, was of unalloyed crucible cast steel made up of many Y-shaped sections, each 12–16 ft long. With such a gun about 450 ft long contg 28 proplnt chambers, it was expected to achieve a muzzle vel of 4500 ft/sec and a range of about 130km (using a proj 8 ft long and weighing 150 lbs)

The gun could lie on the ground, without any carriage, on blocks sloped at a 45° angle. The fin-stabilized arrow proj was inserted in the barrel and the base proplnt chge electrically initiated. As the proj passed the separate Y-pieces, addnl proplnt chges in

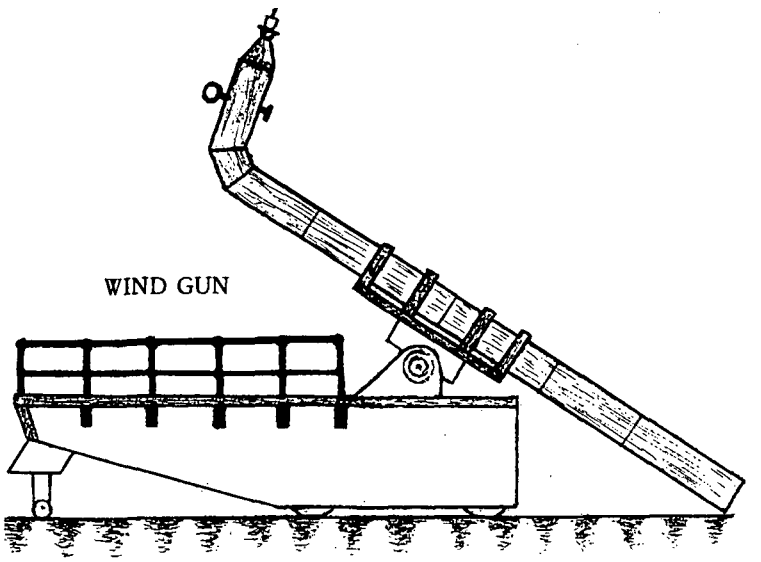


HDP SUPERGUN
VERGELTUNGSWAFFE 3
(V-3)

the side arms were ignited one after another, thus accelerating the vel of the proj as it progressed along the barrel

Another interesting German gun of WWII was *Wind Gun*, developed in Stuttgart. It was designed to shoot a mass of air at an airplane in such a way as to bring it down. The energy for projecting the air was supplied by heat produced on burning a mixt of oxygen and hydrogen. It was claimed that the air shot from this gun could break a 1-inch board at a range of 200 meters, but at longer ranges it was not effective (See drawing)

Ref: L.E. Simon, "German Research in WWII", Wiley, NY (1947), p 180



WIND GUN

It does not seem that other countries participating in WWII developed any original types of guns. Most of them were either of WWI or prior to WWII. Many of the Belgian, Dutch, French, Czech, Norwegian, Polish, Russian and Yugoslavian (and some British) guns were captured by Germans and used by them. They are listed together with regular German Guns in PATR **2510**(1958), pp Ger 237 to Ger 262. They were marked by letter f for French, as for example: 2cm GebFlak(f); i for Ital: 2cm M35(i); p for Polish: 3.7cm Pak (p); r for Russian: 7.62cm KK(r); e for English: 8.38cm FK271(e); b for Belgian: 7.5cm FK(b); j for Yugoslavian: 7.65cm FK(j); h for Dutch (Holland): 7.5cm FK(h); t for Czechoslovakian (Tschekoslovakei): 7.5cm FK17(t); n for Norwegian: 7.5cm FK01(n), etc

For Russian guns of WWII we have good illustrations on seven separate sheets of PATR **2145**(1955). The artillery pieces include items ranging from 25mm Antiaircraft Gun M1940 to 152mm Howitzer 1943 and 160mm Mortar. There are also illustrations of rocket launchers: 82mm, 132mm, M-13 and 300mm M-31. Also included illustrations of Armored Car BA-64; Medium Tank and two Heavy Tanks. Most popular of Russian guns are 76.2mm and they are considered just as good as famous French 75mm guns

Refs: 1) W.W. Greener, "The Gun and Its Development", 9th Edition, Bonanza Books, NY (1910) (Distributed by Publishers Central Bureau, 34 Engelhard Ave, Avenel, NJ 07001) 2) Hayes (1938), 59–370 3) J.R. Newman, "The Tools of War", Doubleday, Doran & Co, NY (1943), 69–175 4) Ohart (1946), pp 3, 5 & 173 5) G.M. Barnes, "Weapons of World War II", VanNostrand, NY (1947), pp 122–26, 128–31, 134–35, 140–42 & 144–56 6) Anon, "Principles of Artillery Weapons", **TM9-3305-1**(1956), 12–17, 169, 173–74) 7) GlossaryOrdn (1959), 142-L 8) Encycl of Expls, PATR **2700**, Vol **2**(1962), p C26-L to C29-R

Gun Ammunition Components. Historical discussion giving mechanical and chemical details on development of these components is in Ref

Ref: H.S. Rentell, JJuniorInstEngrs **54**, 245–57(1944) & CA **39**, 621(1945)

Gun, Atomic. See Note under Gun and under Nuclear Weapons

Gun-Barrel Corrosion Inhibitor. A mixt of mineral oil 0.3, kerosene 0.15, and colloidal graphite 1 kg when applied to small shot, checks the corrosive action of Gunpowder on gun barrels

Ref: Società Faro, ItalP 487073(1953) & CA **50**, 11224(1956)

Gun Breech is the rear part of the bore, especially the opening that permits the projectile to be inserted in the rear of the bore. It is closed, after insertion of round of ammo, by *breechblock*. This usually contains the firing pin and in many types of guns it is also used to chamber the round. The mechanism which unlocks and withdraws the breechblock from the breech is known as *breechblock operating mechanism*. After firing the round the mechanism serves to open the breech for ejection of empty cartridge and for loading a new charge
Ref: GlossaryOrdn (1959), 48-R & 49-L

Gun Carriage is mobile or fixed support for a gun. It sometimes includes elevating and traversing mechanisms. Wheeled carriage was invented ca 1470
Ref: GlossaryOrdn (1959), 148-R

Guncotton. Nitrocellulose of high nitrogen content (13.35 to 13.45%). It is described in Vol **2** of Encycl, under CELLULOSE NITRATE, p C106-R to C108-R

Guncotton Compressed. It was mentioned in Vol **2** of Encycl, p C101-R that E.A. Brown (asst to F. Abel) discovered that dry compressed NC could be made to detonate violently by MF cap, such as A. Nobel had used to detonate NG. The chge was too sensitive and could not be used for military purposes. This discovery took place ca 1869. Brown also discovered later that compressed Guncotton contg 15–20% water also could be detonated by MF cap, provided there was a small intermediate chge of dry compressed Guncotton (principle of *booster*). This permitted the use of large blocks of moist compressed Guncotton quite safely

Russians took advantage of this discovery and started to use compressed Guncotton contg 18% water beginning in 1885 as bursting chges in shells, bombs, mines and torpedoes. They continued using it till after Russo-Japanese War (1904–1905) or even later. This is described in Vol **3** of Encycl, p C491-L. Dr Fedoroff served during WWI on a small Russian destroyer Zavetnyi (350 tons, 27 knots), noticed that warheads of Whitehead torpedoes were charged with moist compressed Guncotton

A very strongly compressed Guncotton, prepd in 1890's by S.P. Vukolov, resembled in its appearance elephant's tusk, and for this reason was called *Slonit*, which means Elephantide. It was used as a propellant (Ref 2, p Rus 21)

Compressed moist Guncotton was not powerful and one of the reasons was its low density of loading (1.0 to 1.1) because there were many air spaces in the block. This was improved after A. Hollings patented a method of prepn giving compressed blocks with density of 1.25. This method and its modification by G. Bell (1903) are described by Worden (Ref 1, p 2168). Another method of block-pressing was proposed in 1905 by A. Musker (Ref 1, p 2174). In 1907 J. Carter patented a method in which the chges were made in longitudinal halves or sections to fit the projectile by compression in molds, where the pressure was exerted in a direction transverse to the axis of the mold. The sections were then united by means of collodion soln, to form a single piece. There was no necessity to use lathe-turning (Ref 1, p 2173)

Methods of manuf of wet compressed Guncotton in Austria and Germany are described in Ref 1, pp 2174–76

The use of compressed, moist Guncotton was discontinued at the beginning of this century, being replaced by PA and TNT
Refs: 1) E. Worden, "Technology of Cellulose Esters", VanNostrand, NY, Vol 1, Pt 3(1921), pp 2160–88 2) B.T. Fedoroff et al, "Dictionary of Russian Ammunition and Weapons", PATR **2145**(1955), p Rus 22

Guncotton Dynamite is a nongelatinous expl invented by Trauzl & Abel in 1867. It was prepd by mixing NG 73, Guncotton 25 & charcoal 2 with 15 parts of w and using it in the wet state. It required a strong Blasting Cap for detonation. A similar expl was *Dualin*, used in the US. These expls may be considered as the oldest Dynamites using an active base

Guncotton Dynamites were brisant expls, but their density was low and they required large bore-holes

With the invention of the Gelatin Dynamites by A. Nobel, the manuf of Guncotton Dynamites was discontinued
Ref: Naoúm, NG (1928), p 282

Gun Flash. Same as Muzzle Flash

Gun Gases in Combat Aircraft. (Prevention of Explosion). Residual gases from functioning machine guns form expl mixts with air. Their expln in the gun compartment of combat-type aircraft may cause considerable damage. The following is a typical percentage compn of gun gas after elimination of water vapor by expansion and cooling: CO_2 14, H 19, CO 52, CH_4 1 & N 14%. Expts indicate the avg value of the lower expl limit to be 11.3% dry gun gas at sea level. The simplest method of reducing the hazard is to provide adequate ventilating air and to distribute it properly
Ref: J.J. Horan et al, AeronautEngRev **11**, No 3, 37–43 (1952) & CA **46**, 7331 (1952)

Gun and Guided Weapon. A discussion of the relative advantages of guns (antitank) and guided weapons is given in Refs. From past experience in the last ten years it seems that the modern arsenal should have both
Refs: 1) Lt Col I.S. Weeks, Ordn **55**, pp 552–55 (1971) 2) Editor's Note on p 486, Ord May-June (1973)

Gun Life Factor (GLF) (British). If the useful life of a gun using Cordite MD (MC) (equal to about 2000 firings for the smaller field guns) is arbitrarily taken as unity (1), then the number of firings of a gun using Cordite Mark I would be only one third (0.33). The values 1, 0.33, etc are called "gun life factors". The factor for Cordite RDB is 1.8, for Cordite SC 0.97, for Cordite "Cooled SC" 1.2 and for British Flashless (various compns) 3.7 to 8.0. Compns of Cordites are given in Vol **3** of Encycl, p C534. See Vol **3**, pp C532-Lff for compn of Cordites

Gun Liquid Propellant of Detroit Controls Corporation consists of mixts of hydrazine, hydrazine nitrate & water for achieving hypervelocities with service wt projectiles
Ref: Detroit Controls Corp, Monthly Progress Rept **33**, "Liquid Propellant Gun Systems", May 1956 (RC-198), Redwood City, Calif

Gun Machine. See Machine Gun

Gun Mantlet is, accdg to definition given by Col G.B. Jarrett of Aberdeen Proving Ground, the main basic support for the tank cannon. It is so designed as not only to afford the elevation and depression of the gun but also it becomes part of the forward armor of the turret. Attached to this mantlet, is the special armor necessary for the gun recoil system

Gunmetal (Bronze). An alloy of Cu 90 and ca Sn 10 parts with 2% Zn, 1% Pb and small quantities of Ni, Sb, Fe & Al. Formerly used for casting cannons
Refs: 1) Hackh's Dict (1944), 392-R
2) CondChemDict (1971), 429-R

Gunnery is the art or practice of using cannons or machine guns either on the ground or in the air (Ref 6). It comprises the following three subjects: a) exterior ballistics; b) interior

ballistics; and c) gun construction (Ref 1) (See also under Ammunition and Weapons in Vol 1, pp A383-L to A391-R. See also under Artillery, Vol 1, p A492-L and Ballistics, Vol 2, p B7-L)
Refs: 1) Ordnance College Woolwich, "Gunnery", HMSO, London, Part 1 (1914) & Part 2 (1911) 2) P.R. Alger, "The Groundwork of Practical Naval Gunnery", US Naval Institute, Annapolis, Md (1915) 3) US Military Academy, West Point, NY, "Artillery", Sept 1950: Chap 1: Gun Design, Chap 2: Gun Construction 4) Encycl Brit, "Naval Gunnery", Vol 11, pp 1–3 (1952) (5) Colliers' Encycl, "Naval Gunnery", Vol 14, pp 414–16 (1957) 6) GlossaryOrdn (1959), 143-R

Gunn's Explosive (Amer). Blasting expl proposed in 1899: Na nitrate 63, soft coal 22 & sulfur 15%. Also patented in England and France
Refs: 1) Daniel (1902), p 365 2) Fedoroff & Clift 4 (1946), 42

Gun Pendulum Test. Same as Ballistic Pendulum Test described in Vol 1 of Encycl, pp VII–VIII and by A. Schmidt in Explosivst 10, 23–34 (1962)

Gun Perforator. Expendable perforator using hollow chges to penetrate a well casing
Ref: L. Spencer, USP 2833214 (1958) & USP 2835215 (1958)

Gun-Plow. French & Fancher of NY State Patented in 1862 (Ref 1), a plow, the hollow metal beam of which could alternate as a tongue to which was hitched a team or a gun to fire a ball or grape ammo at marauders (Ref 2)
Refs: 1) USP 35600 (1862) 2) Anon, Army R&D 2, No 12, 16 (1961)
Note: R&D stands for Research and Development

Gunpowder, Black. Brit for Black Powder, which is described in Vol 2 of Encycl, pp B165-L to B175-R
Addnl Ref: F.E. Pounder of Chemical Inspectorate (Brit), "Contaminants in Gunpowder", March 1963 (PA Library C100713) (Not used as a source of info)

Gunpowder, Brown. See BkPdr Modification, known as *Brown-, Cocoa-* or *Chocolate Powder* described in Vol 2 of Encycl, p B173-L. It is also listed under CANNON PROPELLANT on p C30-L of Vol 2

Gunpowder, Initiation and Thermal Decomposition of. Initiation is due to formation of local hot spots which may be formed by adiabatic compression of air pockets or by friction of grit particles. The necessary hot spot temperature is 130° which suffices to melt the sulfur. The formation of liquid phase is important, and it is suggested that the process starts with the reaction of sulfur with oxyhydrocarbons present. These latter can be extracted by solvents from the charcoal and also provide a liquid phase. A high pressure is necessary for rapid reaction. Development of initial reaction continues until the main reaction, oxidation of carbon by K nitrate, is established. Acceleration of the initial reaction leading up to rapid burning is controlled by the oxyhydrocarbons in the charcoal. High speed camera reveals that the Gunpowder grains burn slowly layerwise. Rapid propagation from grain to grain is probably due to emission of hot spray of molten droplets of K salts
Ref: J.D. Blackwood & F.P. Bowden, Proc-RoySoc (London) **A213**, 285–306 (1952) & CA 46, 10623 (1952)

Gunpowder, White. A mixt of 2 parts K chlorate, 1 pt K ferrocyanide & 1 pt sugar
Refs: 1) CondChemDict (1961), 555-L
2) Ditto (1971), not found

Gun Primer is a device for igniting gun proplnt. It usually consists of an ignition element, a BkPdr booster chge, and an extender tube filled with BkPdr. In smaller calibers it may resemble a fuze primer
Ref: Collective, "Ordnance Explosive Train Designers' Handbook", Naval Ordnance Laboratory Report, **NOLR 1111**(1952), p G2

Gun Proof Tests. Ballistic Tests in guns are described in the following Refs:
1) Anon, "Test Methods for Small Arms Ammunition", Ordnance Proof Manual **ORD-M608-PM**, Frankford Arsenal, Philadelphia, Pa (Jan 1945)
2) Anon, US Army Test and Evaluation Command Materiel Test Procedure, **TECP 700-700**, Aberdeen Proving Ground, Md (Jan 1967); Ordnance Proof Manual Vol 1, "Arms and Ammunition Testing" (Oct 1957) and Interim Pamphlet Nos:

- **20-10** Machine Guns (Feb 1966); MTP 3-2-514
- **30-10** Artillery Cannon (May 1965); Material Test Procedure MTP 3-2-509
- **30-30** Recoilless Weapons (March 1965); MTP 3-2-066
- **30-40** Subcaliber Guns (March 1957); MTP 3-2-518

3) Anon, US Army Materiel Command, "Safety Manual", **AMCR 385-100**, Washington, DC (April 1970), pp 28-13ff (Test Firing Recoilless Weapons and Ammunition)

Gun Propellants, Advanced. A principal development goal in the field of aircraft cannon ammunition has been the development of cool-burning gun proplnts. A new series of cool-burning, high impetus, low molecular weight gun proplnt families, largely based on rocket proplnt technology, have been identified after four years of extensive gun-proplnt development at Eglin AFB. The proplnt families contain either cyclic or linear nitramines as crystalline oxidizers in the proplnt matrix. Table shows the principal linear nitramine compds studied

These systems are thermally and chemically stable and burn with excellent combustion efficiency when fired in a closed impetus bomb. However, they are not yet ready for operational gun application because of the classical nitramine combustion anomaly; this anomaly being that a single n value, in the empirical formula to describe the burning rate of a propellant $r = aP^n$, can no longer be defined over the entire pressure range. The benefits of high energy/low flame temp formulations, elimination of thermal barrel erosion, use of aluminum cartridge cases, and the realization of hypervelocity aircraft cannon cannot be obtd until the nitramine combstn anomaly is obviated
Ref: O.K. Heiney, NatlDef (formerly Ordn) **58**, 152–57, Sept-Oct 1973 (9 refs)

Gun Submachine. See Submachine Gun

Abbreviation	Compound	Formula	Heat of Formation ΔH_F, kcal/mole
TAGED	Triamino guanidinium ethylene dinitramine	$C_4H_{22}O_4N_{16}$	+60
DMDTH	Dimethoxy dinitrazahexane	$C_6H_{14}O_6N_4$	−94
DMED	Dimethylethylene dinitramine	$C_4H_{10}O_4N_4$	−35.8
DHED	Dihydrazinium ethylene dinitramine	$C_2H_{14}O_4N_8$	−40

Guns Using Liquid Propellants are briefly described by A.M. Goodall in ARDE (Armament Research and Development Establishment) Memorandum (P) **31/56**, Fort Halstead, Kent(GtBritain) and previous British Reports which are listed on p 16 of the above Memorandum

GuP or **GuPicr.** Abbrns for Guanidine Picrate

Gurney Constant and Gurney Formulas. *Gurney constant* is a factor for use in Gurney formulas. It is constant for each explosive, but varies with different expls. *Gurney formulas* is a series of formulas, each of them corresponding to a particular geometry of the container, which enables quite accurate prediction of the initial fragment velocity, The velocity is dependent on the geometry, the explosive used and the ratio of the expl charge and metal weight (Ref 1)

The Gurney formula for cylinders is:

$$V_o = \sqrt{2E}\sqrt{\frac{W_c/W_m}{1+0.5W_c/W_m}}\ \text{ft/sec}$$, while for spheres the formula is the same except that 0.6 is used instead of 0.5

In these formulas:

V_o = initial fragment velocity, ft/sec

$\sqrt{2E}$ = Gurney constant, ft/sec

W_c = charge weight, lb

W_m = wt of fragmenting metal, lb

The empirical constant $\sqrt{2E}$ is detd for a particular range of expl to metal ratios. It is expressed as the quantity of energy per unit mass of expl which is available as kinetic energy of fragments. In general, this *energy is somewhat more than half of the energy of detonation.* Rather than reducing the quantities to theoretical terms, the Gurney constants are given as velocities. Following are Gurney constants, $\sqrt{2E}$, expressed in ft/sec for three military expls: 8800 for Comp B, 8300 for Pentolite and 7600 for TNT

The lethality of a fragment is a function of its velocity, weight, and presented area (Ref 2)

Refs: 1) GlossaryOrdn (1959), 143-R & 144-L 2) Anon, "Explosive Trains", US Army Materiel Command Pamphlet **AMCP 706-179**, Jan 1974, p 3-15

Gustavus Adolphus, called in French "Gustave II Adolphe le Grand" (1594–1632), King of Sweden from 1611 to 1632, who introduced many innovations in armament, among them the paper cartridge. He was one of the principal participants of Thirty Year War (1618–1648) and conquered a big portion of the Baltic coast which belonged to Russia and Germany. His victories made Sweden one of the biggest countries of Europe. He was killed at the battle of Lützen leading a cavalry attack

Refs: 1) Claude et Paul Auge, "Nouveau Petit LAROUSSE Illustré", Librairie Larousse, Paris VI^{e} (1953), p 1424-L 2) EncyclBrit **10**(1973), 1048–49

Gustavus Adolphus' Cartridge is described in Vol 4 of Encycl, p D754–55 with Fig 18

Guttapercha (Gummi plasticum or Isonandra gutta), $(C_{10}H_{10})_2$. A group of hydrocarbons (such as fluavil, alban) with volatile oil. It consists of purified and coagulated milky exudates of various trees of *Palaquium* species, native to S. America & Asia, colorless rubber-like masses, sticks or sheets, sometimes yellowish with red streaks; insol in w; partly sol in carbondisulfide, chlf, turpentine and warm benz; softens ca 65 °(Ref 1), melts ca 100°(Ref 2). Used for insulating wires, waterproofing and can be used as a binder in expl compns

Refs: 1) Hackh's Dict (1944), 392-R 2) CondChemDict (1962), 555-R 3) CondChemDict (1971), 767-R (See Note under rubber, natural)

Gutter. A portion of an inner liner of a jet engine which is grooved for better operation

Ref: GlossaryOrdn (1959), 144-L

Guttman, Oscar (1855–1910). German scientist, specializing in expls. Inventor of one of the stability tests for expls. Author of numerous publications and books on expls which were translated into English and other languages. He was killed in an automobile accident in Brussels
Refs: 1) Anon, Ber **43**, 2783(1910) 2) R. Escales, SS **5**, 469–72(1910) 3) M. Giua, "Dizionario di Chimica", U.T.–E.T., Torino, Vol **2**(1949), p 468 4) E. Hatscheck, Chem-Ztg **34**, 845–6(1951)

Guttman's Test. This test for detn of stability of smokeless proplnts is not applicable to those contg NG. In Jannopoulos modification, the finely ground proplnt is kept before the test at 35–38° for 4 days to eliminate w and volatiles. The test is conducted in the manner of Abel's Test (See Vol **1** of Encycl, p A2-L, except that as indicator is a soln of DPhA in sulfuric acid & glycerol). The end point is difficult to observe
Refs: 1) Reilly (1938), 78 2) Kast-Metz (1944), 232 & 309

Gutzeit, Heinrich W. (1845–1888). A German analytical chemist who invented, among other tests, the test for arsenic. The test is performed by adding Zn and dil sulfuric acid to the substance in a test tube, placing on top of it a piece of filter moistened with $HgCl_2$ soln and observing the formation of yel spot on paper, which turns black on standing
Ref: Hackh's Dict (1944), 392-R

Guyton de Morveau, Louis-Bernard (1737–1816). A French lawyer who made chemistry a hobby. He introduced (before Lavoisier), the first chemical nomenclature, promoted the anti-phlogiston theory and translated into French many books. He liquefied ammonia
Refs: 1) Hackh's Dict (1944), 393-L
2) C. et P. Auge, "Nouveau Petit LAROUSSE Illustré, Librairie Larousse, Paris VI^e, 1953, p 1425-R

Gymnastication. Process of artifically exercizing a recoil mechanism by mechanical means in order to prevent undue deterioration in storage. Machine for mechanical exercising is called *gymnasticator.* See also "Exercizer, Recoil Mechanism", in this Vol, p E349-L and in GlossaryOrdn (1959), 144-L

Gypsum (Calcium Sulfate Dihydrate, Alabaster), $CaSO_4.2H_2O$; mw 172.17, sp gr 2.31–2.33, white substance, mp loses $3/2H_2O$ by 128° and rest by 167°, hardness 1.5 to 2. Occurs naturally in the USA, Canada, England, France, Sicily, Switzerland etc; sl sol in w; sol in acids, glycerin and aqueous $Na_2S_2O_3$ and ammonium salts

Since it contains water of crystallization, and because it is cheap and abundant, it was proposed as a temp reducing (cooling) agent in permissible expls (See Hydrated Explosives). It was also previously used as a source of sulfur and for the prepn of sulfuric acid. Many other uses are listed in Refs 3 & 5
Refs: 1) Mellor **3**(1923), 760–64 2) Marshall **3**(1932), 12 & 17 3) CondChemDict (1961), 556 4) Kirk & Othmer **4**(1964), pp 16–21
5) CondChemDict (1971), 430-L

Gyro. Short for gyroscope or gyroscopic

Gyroscope. A device which utilizes the properties of a spinning mass to remain fixed in space. It can be used in a flight instrument which holds the position and indicates deviation from the desired heading. Also used in an autopilot or bombsight mechanism that provides directional stability. In a mechanism called "gyrostabilizer", the vertical motion of a tank gun is stabilized regardless of the pitched motion of moving tank
Refs: 1) GlossaryOrdn (1959), 144
2) OrdTechTerm (1962), 149 3) EncyclBrit **20**(1973), 1080–84

Gyroscopic Action (Kreiseleffekt, in Ger). Concerning the possibility of explaining the drift of an elongated shell in rotation by gyroscopic action, a calculation was undertaken in 1839 by S.D. Poisson. But G. Magnus was the first in 1852 to show the main phenomena, by experiments on models in a lab (Refs 1 & 2)
Refs: 1) C. Cranz & K. Becker, "Exterior Ballistics", His Majesty's Stationary Office, London, Vol 1 (1921), p 325–32 (English)
2) C. Cranz, "Lehrbuch der Ballistik", J. Springer, Berlin, Vol 1 (1925), p 342

Gyroscopic Movement of Projectiles. The English theory of gyroscopic projectile flight was modified to take into account the variation of air resistance and the moment of this force with the "yaw" or obliqueness of a projectile in flight (Refs 1 & 2)
Refs: 1) F. Burzio, Rivista di Atiglieria e Genio **1933**, March, p 119. Its French translation: "Sur le coefficient expérimentaux du movement gyroscopic des projectiles" is in MAF **13**, 281–90 (1934) 2) R. Sutterlin, "Sur quelques developments nouveaux de la théorie gyroscopique", MAF **17**, 199–250 (1938)

DEPARTMENT OF THE ARMY
UNITED STATES ARMY
ARMAMENT RESEARCH, DEVELOPMENT AND ENGINEERING CENTER
PICATINNY ARSENAL, NEW JERSEY 07806-5000

AMSRD-AAR-MEE

28 JAN 2009

MEMORANDUM FOR Defense Technical Information Center, ATTN: DTIC-OQ
(Mr. Larry Downing), Ft. Belvoir, VA 22060

SUBJECT: Distribution Limitation Change

1. Request the distribution limitation statement be upgraded from Statement A (Approved for Public Release) to Statement C (U.S. Government Agencies and Their Contractors). Reason: Administrative/Operational Use, effective 23 January 2009 for the following documents:

AD422747	ADA011845
AD257189	ADA019502
AD274026	ADA057762
AD653029	ADA097595
AD745472	ADA134347
AD768069	

2. Organization requesting this change is:

Commander
U.S. Army RDECOM-ARDEC
ATTN: AMSRD-AAR-MEE
Bldg. 321
Picatinny Arsenal, NJ 07806-5000

3. Any questions please contact the undersigned at (973) 724-4287 or ross.benjamin@us.army.mil.

ROSS C. BENJAMIN
Director, Energetics, Warheads
& Manufacturing Technology
Directorate, METC

DEPARTMENT OF THE ARMY
UNITED STATES ARMY
ARMAMENT RESEARCH, DEVELOPMENT AND ENGINEERING CENTER
PICATINNY ARSENAL, NEW JERSEY 07806-5000

AMSRD-AAR-MEE 16 April 2009

MEMORANDUM FOR Defense Technical Information Center, ATTN: DTIC-OQ
(Mr. Larry Downing), Ft. Belvoir, VA 22060

SUBJECT: Recinding Distribution Limitation Change Request dated 23 Jan 09

1. After further review, I am recinding my request dated, 23 Jan 09, SUBJECT: Distribution Limitation Change Request. The below documents should remain with distribution Statement A (Approved for Public Release):

AD422747	ADA011845
AD257189	ADA019502
AD274026	ADA057762
AD653029	ADA097595
AD745472	ADA134347
AD768062	

2. Rationale: The change was not advisable since the documents have already been in the public domain (distribution statement A) for many years and the information is widely available.

3. Any questions please contact the undersigned at (973) 724-4287 or ross.benjamin@us.army.mil.

Ross C. Benjamin

ROSS C. BENJAMIN
Director, Energetics, Warheads
& Manufacturing Technology
Directorate, METC